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A LABORATORY MANUAL
OF
INORGANIC CHEMISTRY

By JOHN B. EKELEY, Ph.D., Sc.D.
Professor of Chemistry, University of Colorado

TO ACCOMPANY

A TEXT BOOK OF INORGANIC CHEMISTRY

By A. F. HOLLEMAN, Ph.D., F.R.A.Amst.
Professor Ordinarius of Chemistry, University of Amsterdam

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FIRST THOUSAND

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PREFACE

The author has used Professor Holleman's "Text-Book of Inorganic Chemistry," in his classes for a number of years, and each year the merit of this splendid text becomes more apparent. To meet a demand in his own classes for a laboratory manual written and arranged for use in connection with Professor Holleman's text, he has gathered together the set of experiments here published. He has found them to work well in his own laboratory, and hopes that they will prove a help to others in the teaching of beginners in chemistry.

The author wishes to acknowledge his indebtedness to Professor Holleman, to Professor H. C. Cooper, Syracuse University, and especially to Professor Harry A. Curtis, University of Colorado, for many valuable suggestions.

Boulder, Colorado,
June, 1912.

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LABORATORY MANUAL OF INORGANIC CHEMISTRY

INTRODUCTION

Experiment 1. The Bunsen Burner. After igniting the gas from a Bunsen burner, vary the quality of the flame by alternately opening and closing the air passages at the bottom of the burner. What is the appearance of the flame when the air passages are closed? When they are open? Which flame would be better for heating a liquid in a glass vessel? Why? The subject of flame will be studied in detail later.

Experiment 2. Glass Tubing. Cut a piece of glass tubing of about 4 mm. bore and 15 cm. long. To cut glass tubing at a given point, a sharp transverse scratch is made by means of a good triangular file and the tube is broken at this point. To obtain a clean break, grasp the tube so that the thumbs are opposite to and near the scratch. Then bend the tube away from the scratch, at the same time pulling slightly.

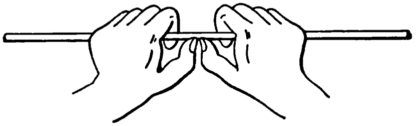
FIG. 1.

Attach a "wing top" to your Bunsen burner. Using the luminous flame, hold, with both hands, the glass tube lengthwise in the flame. Rotate the tube slowly on its axis. When the tube has softened so that it will bend from its own weight, remove it from the flame, and bend it quickly to a right angle. Bend thus several tubes to various angles. Fire polish the ends of the tubes by turning them in the blue flame of the Bunsen burner.

For glass tubing too large to be cut as described above, another method is used. Make the file scratch as before. Fold two pieces of filter paper into a straight ribbon of 10 or 15 cm. length. Wet them and wrap them around the tube on opposite sides of the scratch and about 2 mm. from it, so that there is a uniform ring of uncovered glass between the papers. Then let a small, pointed flame from the blast lamp play upon the uncovered ring, at the same time turning the tube slowly upon its axis. In a few moments the tube will fall apart at the scratch.

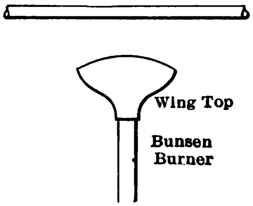

FIG. 2.

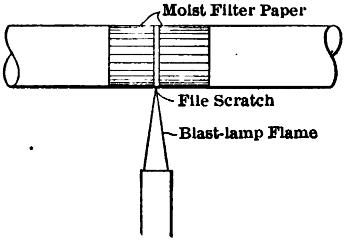

FIG. 3.

To draw a tube out to capillary size, hold it in the flame of the Bunsen burner until it is soft. Remove the tube from the flame and draw it out slowly to the required size.

A tube may be closed at one end by rotating it in the flame as in fire polishing, the operation being carried on until the end falls together of its own weight. If it is desired to make a closed tube from a longer tube, heat the tube at the proper point in a burner or blast lamp flame, at the same time rotating it slowly. When the glass is soft, pull the tube out to capillary dimensions and allow the hottest part of the flame to cut the capillary. Heat the roughly closed end again until it becomes soft, remove it from the flame, and blow it out to a symmetrical form.

Practise the above exercises until you have acquired some skill in their manipulation.

Experiment 3. The Cork Borer. The cork borer is a sharpened brass tube. Bore a hole half way through a good cork. Finish the hole by boring another from the opposite end so that the two holes meet exactly. The hole may be smoothed out by means of a rat tail file. Practise boring holes to fit tubing of various sizes.

Experiment 4. Making a Wash Bottle. Fit a good cork to a liter flask. Cut two pieces of glass tubing of 4 mm. bore. Bend them to the form shown in Fig. 4. Draw another tube out to capillary dimensions and cut it to a total length of 4 cm. Fire polish all ends. Fit the parts together as in the diagram, using a piece of rubber tubing to connect up the nozzle (the small tube with capillary). The whole apparatus is called a " wash bottle."

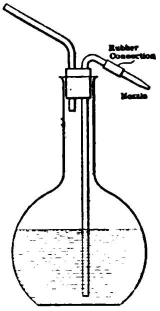

FIG. 4.

PHYSICAL AND CHEMICAL PHENOMENA

*¶ 6

Experiment 5. (*a*) Hold a platinum wire in the blue Bunsen flame. In what respect has the wire changed? Is the change temporary or permanent? Would you call the change physical or chemical? By means of a forceps hold a piece of magnesium wire in the flame until it emits a bright white light. Note the condition of the wire on removing it from the flame. In what respect is it different from what it was before? Is the change temporary or permanent? Physical or chemical?

---

* The paragraph numbers refer to the corresponding paragraphs in Holleman's "A Text-Book of Inorganic Chemistry."

(*b*) Place a few crystals of naphthalene in a test tube. Heat the tube gradually until the naphthalene boils. After the naphthalene has cooled, remove it from the tube and compare it as to color, odor, etc., with naphthalene you have not heated. Was the change brought about by the heat physical or chemical?

Now perform the same experiment using sugar instead of naphthalene. Describe the changes which take place. Is the change physical or chemical?

(*c*) Obtain from your instructor an electric cell and a galvanoscope. Connect the poles of the cell with the binding posts of the galvanoscope. Has the wire coil of the galvanoscope acquired any new property? Is the change physical or chemical?

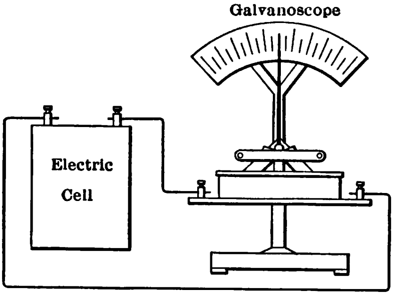

FIG. 5.

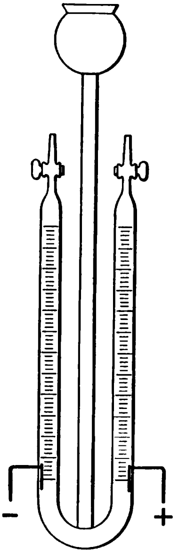

FIG. 6.

Now obtain from your instructor a Hofmann's apparatus for the decomposition of water. Connect the poles of the cell with the electrodes of the apparatus. What is the origin of the gases formed? Has a physical or a chemical change taken place?

CHEMICAL OPERATIONS

¶ 7

Experiment 6. Solution. (*a*) Grind to a fine powder about 15 g. of potassium bichromate in a mortar. To a 100-c.c. flask add about 50 c.c. distilled water. Add the powder to the water as long as it will dissolve, shaking the whole occasionally. After about five minutes, take the temperature of the solution. Measure out into a dry, weighed porcelain dish, by means of a pipette, 25 c.c. of the solution, evaporate it to dryness on the water-bath,* and weigh it again. How

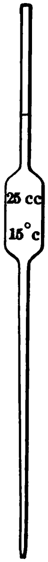

Fig. 7.

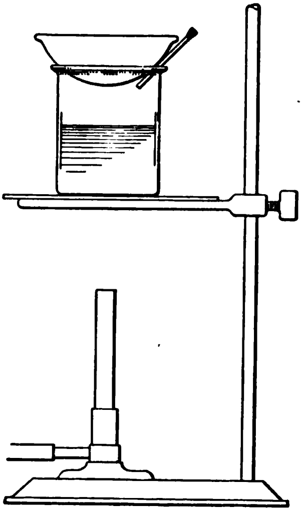

Fig. 8.

---

* A beaker containing boiling water may serve as a water-bath, a porcelain dish, containing the evaporating liquid, resting upon it with pieces of broken matches placed between the dish and the rim of the beaker to allow the steam to escape.

many grams of potassium bichromate dissolved in 25 c.c. of water at the temperature of the experiment? Calculate the solubility of potassium bichromate in a liter of distilled water at this temperature.

Repeat the experiment, using a temperature about 20° higher, and calculate the solubility to 1 l. How is the solubility of potassium bichromate related to the temperature?

(*b*) To three test tubes, containing about 1, 2, and 3 c.c. of water, add respectively 3, 2, and 1 c.c. of alcohol. Is there any limit to the solubility of alcohol in water or of water in alcohol?

(*c*) Try to mix a few drops of ether with water. Is ether readily soluble in water?

(*d*) Pass hydrogen sulphide (see instructor) into 100 c.c. of water in a 200-c.c. beaker for about three minutes. After blowing the air out of the top of the beaker, test the liquid for any odor. Now boil the liquid (under the hood) for about ten minutes. Test it again for odor. Is hydrogen sulphide soluble in water, and, if so, how is the solubility related to the temperature?

Experiment 7. Separation of a Solid and a Liquid. Fold a circular filter paper (of about 9 cm. diameter) on one diameter. Then fold it again at a right angle to the first fold. Open the filter so as to form a cone, and place it in a funnel. Moisten the filter with a stream of water from your wash bottle, and fit it carefully to your funnel. To 20 c.c. of a hot dilute silver nitrate solution in a 100-c.c. beaker, add drop by drop a dilute sodium chloride solution until no further white solid substance (precipitate) forms.* The point at which all is precipitated may be determined by boiling the solution for a few minutes and then letting a small quantity of the sodium chloride solution run down the side of the beaker and noting whether it remains clear as it descends. Now stir the solution, and boil it until the precipitate settles to the bottom and the supernatant liquid is clear. Hold a stirring rod against the lip of the beaker and pour the clear liquid on to the filter (decantation), catching what runs through (filtrate) in a clean beaker. In this manner, pour off the liquid as closely as possible. To the filtrate, add a drop of silver nitrate solution. Does a precipitate form? Now add about 30 c.c. of hot distilled water to the solid in the original beaker, and filter again, using the original filter, but a new beaker under the funnel (washing by decantation). On testing the filtrate as before, does a precipitate form? Continue the operation until only a slight cloudiness is shown. Then, by means of a stream of hot water from the wash bottle, wash all the precipitate on to the filter.

---

* When a sodium chloride solution is added to a silver nitrate solution, a solid, insoluble substance, silver chloride, is formed, while a soluble substance, sodium nitrate remains in the solution. The excess of sodium chloride over that necessary to precipitate the silver chloride also remains in the solution. Its presence in the filtrate may be detected as above by means of silver nitrate solution.

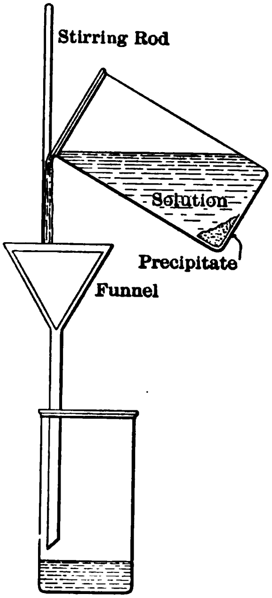

FIG. 9.

If the precipitate were pure, would a cloudiness form on testing the wash water? Now wash the precipitate by playing a stream of water upon it, beginning at the top of the filter paper and following it around in a spiral until the bulk of the precipitate is reached. Then stir the precipitate up by means of the stream of water. Catch the filtrate in a test tube, and test it with a drop of silver nitrate solution. Continue the operation until the filtrate remains clear on testing. What can you say regarding the purity of the precipitate?

After the experiment is finished, place the silver chloride in the bottle provided for the preservation of silver residues.

Experiment 8. Crystallization. Dissolve about 20 g. of crude potassium bichromate in the least possible amount of boiling water. Filter the solution and, after removing a small quantity of it, set the filtrate aside to crystallize. To the small quantity removed, add 5 c.c. concentrated hydrochloric acid and then a little barium chloride solution. Does a white precipitate form? * After allowing the original filtered solution to stand until a considerable quantity of crystals have separated (a day or so), pour off the mother-liquor from the crystals that have formed. Dry the crystals by pressing them between clean filter paper. Dissolve a few crystals in water and test the solution as before with hydrochloric acid and barium chloride. If a white precipitate forms, dissolve the whole mass of crystals in a minimum quantity of boiling water and recrystallize them. Repeat the operation until, on testing with barium chloride, no white precipitate forms.

Experiment 9. Distillation. (*a*) To about 100 c.c. of water in a 200-c.c. Florence flask, add a small quantity of powdered potassium permanganate. Fit the flask with a one-holed cork carrying a delivery tube bent to an acute angle

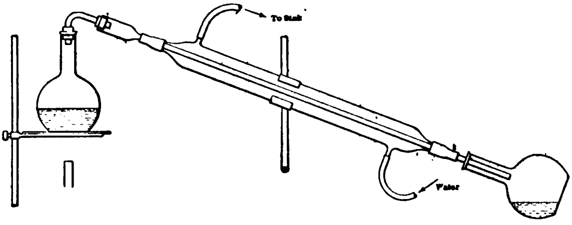

FIG. 10.

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* Crude potassium bichromate contains as an impurity potassium sulphate, the presence of which is detected by the above process.

of 60° (see Exp. 2). Connect the flask with a Liebig condenser as in Fig. 10, and distil the liquid, catching the distillate in a clean flask. What is the distillate?

(*b*) Take the specific gravity of some crude spirit by means of a small hydrometer.* Distil the spirit and take the specific gravity of the distillate. If the purity of the alcohol increases as the specific gravity decreases, what can you say concerning the relative purity of the distilled and the undistilled samples?

Experiment 10. Sublimation. Place about 3 g. of commercial ammonium chloride in a porcelain dish. Cover the dish with a beaker containing cold water. Heat the dish gently until the beaker is covered with the sublimed ammonium chloride. Compare the original product with that obtained by sublimation. Which appears to be the purer?

THE ELEMENTS

¶ 8

Experiment 11. Separation of Gunpowder into its Constituents. Pour about 20 c.c. of water upon about 10 g. of common gunpowder in a 100-c.c. beaker and warm the whole gently. Filter off the undissolved black mass, and allow the filtrate to evaporate to a small volume on a water-bath, and finally set it aside to crystallize. Compare the white crystals thus obtained with others known to be saltpeter. Wash the black residue on the filter two or three times with a stream of hot water from a wash bottle. After the filter is *completely dry,* shake the black substance into 10 c.c. of carbon bisulphide in a small beaker. Keep the carbon bisulphide away from the flame, since it is very inflammable. Finally filter off the black residue through a *dry* filter and compare it with charcoal powder. After the carbon bisulphide has evaporated from the filtrate, compare the precipitated crystals with sulphur. Are they sulphur crystals? Of what substances is ordinary gunpowder composed? What do we call substances, like carbon and sulphur, which we have not been able to decompose into simpler constituents?

---

* If you are not familiar with this method of taking specific gravities, look the subject up in a test book on physics.

Heat a little saltpeter in a hard glass tube, using the blast lamp, and hold a glowing splinter in the tube. Is a gas given off? Heat the saltpeter until no more gas is given off (this will require about five minutes). After the heated mass has cooled, add a few drops of sulphuric acid to the tube. Is a red gas given off? Try in the same way a little saltpeter which has not been heated. Is saltpeter changed on being heated strongly? Would you class saltpeter with carbon and sulphur?

OXYGEN

¶ 9

Experiment 12. Preparation of Oxygen. (*a*) In hard glass test tubes, heat small quantities of mercuric oxide, potassium chlorate, manganese dioxide (blast lamp), and lead peroxide, testing by means of a glowing splinter any gas that may be given off.

(*b*) Warm gently a little potassium bichromate with 2 c.c of concentrated sulphuric acid in a test tube and test for oxygen.

(*c*) A common laboratory method for the preparation of oxygen is as follows: Mix well about 5 g. of powdered potassium chlorate with 2.5 g. of powdered manganese dioxide which has been dried by heating it a few minutes in a porcelain crucible. Fit a hard glass tube with a one-holed cork stopper carrying a delivery tube. Clamp the tube in a horizontal position and heat the chlorate mixture. Collect the oxygen by displacement of water in inverted cylinders. Take the delivery tube out of the water before removing the flame from the heated mixture. Why? Cover the cylinder with a ground-glass plate before removing it from the water. See Fig. 11.

Experiment 13. (*a*) **Physical Properties of Oxygen.** Record the color, odor, taste, state of aggregation, etc., of the element oxygen.

Weight of a Liter of Oxygen at 0 deg. 760 mm. Clean y * thoroughly a 500-c.c. flask. Fit the flask with a two- rubber stopper containing an entrance tube reaching to ottom of the flask and bent to a right angle just outside e stopper. Fit the other hole with a similar exit tube

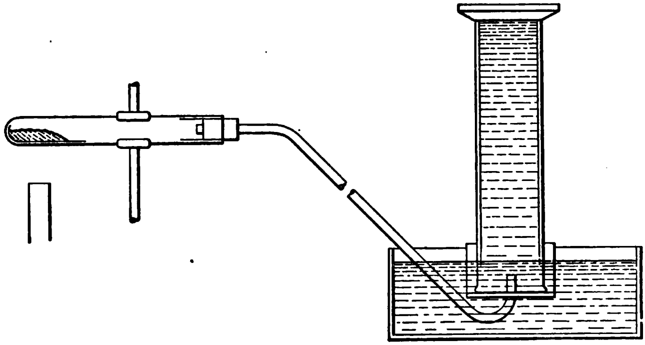

FIG. 11.

reaching only through the stopper. Fit two pieces of good rubber tubing on the tubes so that the connections are air-tight. Close the two rubber tubes by means of good pinch-cocks. Suspend the flask by means of a wire or string to one arm of a good balance and weigh it to .01 g. Pass a stream of oxygen slowly through a Woulff bottle containing concentrated sulphuric acid and then through the flask, letting the gas enter through the long tube. After oxygen has passed through the apparatus for about five minutes, disconnect the flask from the Woulff bottle and immediately close the rubber tubes by means of the pinch-cocks. Then weigh the flask again very carefully to .01 g. Take the reading of the barometer, and the temperature of the room in the neighborhood of the flask. Now fill the flask completely with water and weigh it, together with the connections, on an ordinary balance. The weight in grams of the flask full of water minus the weight of the flask empty gives the volume of the flask in cubic centimeters. Why? Calculate * the weight of a liter of oxygen at normal temperature and pressure as follows:

---

* To dry a flask quickly, rinse it with a little alcohol, and then draw air through it, at the same time warming the flask gently.

Weight of flask full of air.............. $=a$..........g.
Weight of flask full of oxygen........... $=b$..........g.
Volume of the flask .... $=c$..........c.c.
Temperature........................... $=d$..........°
Pressure .... $=e$..........mm.

Then b minus a is the excess in weight of c c.c. of oxygen above that of c c.c. of air, at d° and e mm. Since the volume of a gas varies inversely as the pressure and directly as the absolute temperature, the volume, V, at 0° and 760 mm. will be

$$\frac{273\,e\,c}{760(273+d)}\text{ c.c.}$$

At 0° and 760 mm. 1 c.c. of air weighs .001293 g. Then V c.c. will weigh V multiplied by .001293 g. The same volume of oxygen will weigh $V \times .001293 + (b-a)$ g., and 1 l. of oxygen at 0° 760 mm. will weigh

$$\frac{1000[.001293V+(b-a)]}{V}\text{ grams.}$$

---

* See the next paragraph for the methods of calculating gas volumes, etc.

CALCULATIONS CONCERNING VOLUMES AND WEIGHTS OF GASES

Experiment has shown that the volume of a gas varies directly as the absolute temperature (Law of Charles) and inversely as the pressure put upon it (Law of Boyle).

The pressure is measured by means of a barometer. The following is a crude form of the instrument: A glass tube closed at one end, and about 800 mm. long, is completely filled with mercury. The thumb is then placed over the open end of the tube, the whole inveretd, and the open end placed in a vessel of mercury. On removing the thumb, the mercury falls in the tube to a height corresponding to the pressure of the air at the time of the experiment. This pressure per square centimeter is the weight of mercury supported by the air in a tube one square centimeter in cross-section. At sea level, at 0° C., this pressure does not vary much from 1033 g. per square centimeter, or the weight of a column of mercury 760 mm. high. This is called the "normal pressure." When we speak of the normal temperature and pressure, we mean 0° C. and 760 mm. pressure.

The temperature is measured by the centigrade thermometer, whose scale has 0° as the freezing point of water and 100° as the boiling point under a pressure of 760 mm.

When the temperature of a gas is increased 1°, the volume increases 1/273 of what the volume of the gas would have been at 0°. If the gas is cooled 1°, the volume decreases by a corresponding amount. If then a gas at 0° could be cooled 273° according to this law, the volume would be nothing. This is evidently impossible. The gas could never reach this temperature, since liquefaction would occur before the temperature 273° below zero was reached. This consideration, together with others which cannot be discussed here, point to a temperature of 273° below 0°, at which there is an absence of heat. This point is called the "absolute zero" of temperature.

If V is the volume of a gas, P its pressure, and T its absolute temperature, then

$$V \text{ varies as } T/P, \text{ or}$$
$$V : V' :: T/P : T'/P'$$

in which V is the volume of the gas under the pressure P and the temperature T, and V' the corresponding volume at the pressure P' and the temperature T'. If t is the centigrade temperature, the above formula would become

$$V : V' :: \frac{273+t}{P} : \frac{273+t'}{P'}, \text{ since } T = 273+t.$$

Example. What volume would a gas, whose volume was 300 l. at 720 mm. and 20°, occupy, if the pressure and temperature were changed to 840 mm. and 30° respectively?

Let V' in the above formula represent the known volume at the pressure P' and the temperature T', and let V represent the required volume at the pressure P and the temperature T. Then

$$V : 300 :: \frac{273+30}{840} : \frac{273+20}{720},$$

$$V = 265.91 \text{ l.}$$

Solve the following problems:

1. A gas has a volume of 1 l. at 10° and 760 mm. What would its volume be at 50° and 700 mm.?

2. A gas has a volume of 200 c.c. at 25° and 729 mm. At what pressure would the volume be doubled, if the temperature were changed to 0°?

3. At what temperature would a gas which occupies 500 c.c. at 25° under a pressure of 600 mm. be doubled, if the pressure were changed to 800 mm.?

Problems concerning the weights of a given volume of a gas under different conditions of temperature and pressure may be solved in a similar manner by use of the following formula, which expresses the fact that the weight, W, of a given volume of gas varies directly as the pressure, P, and inversely as the absolute temperature, T:

$$W : W' : : P/T : P'/T'.$$

Problem. If 1 l. of air at 0° and 760 mm. pressure weighs 1293 g., what would a liter of air weigh at 30° and 700 mm.?

Law of Henry. The weight of a gas dissolved in a solvent with which it does not unite chemically, varies directly as the pressure.

Problem. 100 l. of water dissolve 3192 g. of oxygen at 0° and 760 mm. What would the solubility be at 340 mm.?

Experiment. 14. Chemical Properties of Oxygen. (*a*) Plunge a glowing splinter into a jar of oxygen.

(*b*) Fasten a small piece of charcoal to a wire about 15 cm. long, heat it until it glows, by holding it in a flame for a short time, then plunge it into a jar of oxygen. When the glowing ceases, remove the charcoal and pour a few cubic centimeters of clear lime-water into the jar. The turbidity of the lime-water, which results upon shaking the jar, is a test for the presence of carbon dioxide, a gaseous substance always formed when carbon burns in sufficient oxygen.

(*c*) Ignite a piece of sulphur in a deflagrating spoon and lower it into a jar of oxygen. Has the gaseous substance, formed by the burning of the sulphur, any odor? Pour a few cubic centimeters of water into the jar, and plunge a strip of blue litmus paper into the water. Observe whether or not the paper changes color. Taste a drop of the water. The above tests are characteristic of a gaseous substance, sulphur dioxide, always formed when sulphur burns in oxygen.

(*d*) Burn a piece of phosphorus about the size of a pea in a deflagrating spoon in a dry jar of oxygen. The jar is filled with oxygen by displacement of air. Ignite the phosphorus by means of a hot glass rod. **In handling phosporus, always use forceps. Never pick it up with the fingers, for a phosphorus burn heals with difficulty.** Note the white powder which forms as the phosphorus burns. Add a little water, taste a drop of the solution, and test the remainder with litmus paper. The white product formed by the burning of phosphorus in oxygen is called phosphorus pentoxide.

(*e*) Have ready a jar of oxygen. Straighten out a piece of watch spring * about 20 cm. long by passing it through the blue Bunsen flame. Push a knife blade through the center of a piece of cardboard which is large enough to cover the mouth of the jar. Insert the spring through the cardboard. Wrap a little twine around one end of the spring, and dip it into a little molten sulphur. After igniting the sulphur, plunge it into the jar of oxygen, and, after the iron begins to burn, feed it gradually into the jar as long as combustion continues. The black ash is an oxide of iron.

Experiment 15. Proof that the Products of Oxidation Weigh more than the Substance before Oxidation. Place about 5 g. of clean copper turnings in a small, clean crucible and weigh the whole very carefully. Heat the crucible and turnings on a clay triangle at red heat for five minutes. Let it cool and weigh it again. Have the crucible and its contents increased in weight? To what is the change due? What is the appearance of copper after heating?

* An iron wire may be used instead of a watch spring.

HYDROGEN

¶¶ 11–13

Experiment 16. Preparation of Hydrogen. (*a*) Repeat Exp. 5. *c*, second part.

(*b*) Fit a 500-c.c. flask with a one-holed cork carrying a delivery tube. Place about 20 g. of mossy zinc in the flask, and add 200 c.c. of hydrochloric acid (1:1). Collect the gas by displacement of water.

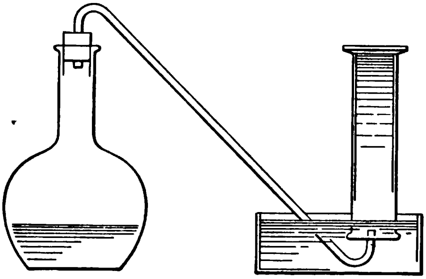

Fig. 12.

(*c*) Perform the same experiment using aluminum turnings and a dilute solution of potassium hydroxide.

(*d*) Wrap a piece of lead foil loosely around a piece of sodium the size of a pea. Drop the sodium into a basin of water, and collect the gas that is generated.

(*e*) Place about 200 g. of coarse iron filings in the middle of an iron gas pipe supplied with corks carrying entrance and exit tubes. Heat the gas pipe by means of a blast lamp several Bunsen burners, or a suitable furnace. Pass steam through the hot pipe. Collect the hydrogen over water.

Experiment 17. Hydrogen Lighter than Air. Weight of a Liter of Hydrogen. ¶ **12.** Fit a dry * 500-c.c. flask with a two-holed stopper and connections as in Exp. 13, *b*, and weigh it carefully to .01 g. Generate hydrogen by means of zinc and dilute hydrochloric acid. Placing the flask in an inverted position, fill it with the gas, which has been dried by passing it through a Woulff bottle containing concentrated sulphuric acid. When the flask is full, take special pains in having all the connections air-tight, and weigh it to .01 g. Find the volume of the flask as in Exp. 13, *b*. Take the reading of the barometer and the temperature of the room at the time of the experiment. Calculate the weight of a liter of hydrogen at 0° and 760 mm. pressure, remembering that the loss of weight observed is the amount by which the weight of the flask filled with air exceeds its weight when filled with hydrogen.

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* See foot-note under Exp. 13 (*b*).

Experiment 18. Chemical Properties of Hydrogen. ¶13. (*a*) Hold a cylinder of hydrogen in an inverted position and ignite the gas.

(*b*) Hold a cylinder of hydrogen in an inverted position and insert a blazing splinter into the gas. Is hydrogen a supporter of combustion?

(*c*) Collect half a cylinder of hydrogen by displacement of water. Remove the cylinder from the trough, and allow the water to run out. After a few minutes the air and the hydrogen will have mixed. Ignite the gas mixture, holding the open end of the cylinder down and away from the face.

(*d*) Using a wide-mouthed cylinder, perform a similar experiment using oxygen instead of air.

(*e*) **Reduction of Copper Oxide.** Fire polish the ends of a glass tube of about 1 cm. diameter and 25 cm. long. Fit it with one-holed corks, one containing a glass tube about 3 cm. long and 5 mm. bore. Place about 5 g. of black copper oxide in the middle of the tube in an even layer. Connect one end of the reduction tube with a rubber tube about 60 cm. long, and the other with a U-tube filled with granular calcium chloride. Connect the calcium chloride tube with a hydrogen generator, and allow the gas to pass through the apparatus until all the *air is expelled.** Now heat the glass tube containing the oxide, heating it carefully at first so as not to break the tube. Note carefully what occurs in the tube. What collects at its farther end? When the action has ceased, allow the tube to cool, while the hydrogen is still passing through it.

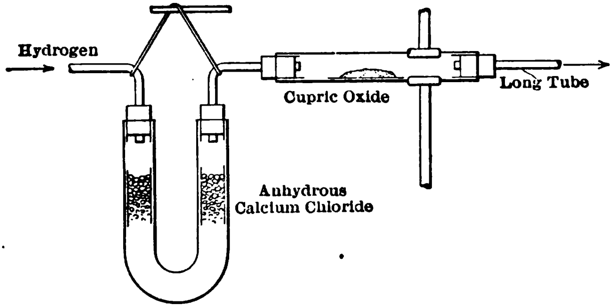

FIG. 13.

When all is cool, disconnect the tube, and remove the substance. Place some pieces of the material on a sheet of paper and press it with a knife blade. What is it? Explain the reaction.

THE CONSERVATION OF MATTER

¶ 14

Experiment 19. Weigh out .5 g. of c.p. potassium sulphate and .75 g. of c.p. barium nitrate. Dissolve the salts each in about 20 c.c. of distilled water and bring the solutions to boiling. After adding 2 c.c. of nitric acid to the sulphate solution, add the nitrate solution very slowly, stirring the mixture with a glass rod. Rinse the beaker with a stream of hot water so as to bring the last traces of nitrate into the solution. Allow the precipitated solution to stand until the next period. Then decant the clear supernatant liquid as closely as possible through a good filter, catching the filtrate in a clean beaker. Wash the precipitate twice with 30 c.c. of water by decantation. Then bring all upon the filter with a stream of water from the wash bottle, and wash the precipitate three times upon the filter. Evaporate the filtrate to dryness on the water-bath in a weighed porcelain dish. When the whole is cool, weigh to .01 g. Place the filter paper containing the precipitate in a weighed porcelain crucible, and heat the whole upon a triangle about 10 cm. above a low Bunsen flame. When the moisture is driven off, heat the crucible gradually with a hotter flame, and finally with the full flame. When the precipitate is snow white, cool and weigh it. It is necessary that none of the precipitate or filtrate be lost, hence extreme care must be taken in all the operations included in the experiment.

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* To determine if all air is expelled, collect a test tube full of the gas over water and ignite it. If the gas burns quietly without "whistling," the copper oxide may be safely heated.

Allowing for unavoidable errors, what can you say regarding the relation of the weights of the factors of the chemical change to those of the products?

WATER

¶¶ 15–19

Experiment 20. Have ready a hydrogen generator. Pass the stream of hydrogen first through a U-tube filled with granular calcium chloride (why?), and then through a glass tube drawn out to an opening about 1 mm. in cross-section. When all the air has been driven out of the apparatus (see note under Exp. 18, *e*), ignite the hydrogen. Hold a cold, dry glass jar over the flame, and note what forms on the inner surface of the jar.

Experiment 21. Water of Crystallization. Heat a few crystals of copper sulphate in a test tube. What collects on the upper, inner surface of the tube? Try some crystals of barium nitrate in the same way. Is there any change?

Experiment 22. Conductivity of Water. ¶ 15. Procure from your instructor a storage cell, a decomposition cell, and a galvanometer. Fill the cell with water containing a little common salt in solution, place it in the circuit with the storage cell and the galvanometer. Take the reading of the instrument. Repeat the experiment, using tap water, after carefully rinsing the decomposition cell. Repeat a third time, using distilled water. Compare the three readings of the galvanometer with one another. If you could obtain absolutely pure water, do you think that it would conduct the electric current?

Experiment 23. Purity of Water. ¶ 17. Evaporate on a watch glass a few c.c. of water from the tap. Does the water contain impurity? Now distil some of the tap water (see Exp. 9) and evaporate a few c.c. of the distillate. Compare the tap water with the distilled water as to purity.

Experiment 24. Common Salt in Ordinary Water. ¶ 17. A solution of silver nitrate added to water which has been made acid with nitric acid is a test for chlorides in water. Test a specimen of tap water in this way. If a white cloudiness forms, the water contains chlorides.

Experiment 25. Composition of Water by Volume. ¶ 18. Repeat Exp. 5 *c*, second part, and measure carefully the volumes of the gases.

Experiment 26. Composition of Water by Weight. ¶ 18. Prepare a glass tube about 25 cm. long and 1 cm. in diameter and weigh it to .01 g.* Place about 1 g. of dried copper oxide in the tube and weigh the whole to .01 g. Fill a U-tube with granular calcium chloride and fit it with one-holed rubber stoppers containing entrance and exit tubes bent at right angles. Fit the entrance and exit tubes with rubber tubes about 2 cm. long plugged at one end with closed glass tubes. Weigh the U-tube to .01 g. Support one end of the tube containing the copper oxide by means of a clamp on a ring stand and connect the tube to an apparatus for producing dry hydrogen (Kipp's generator and Woulff bottle containing concentrated sulphuric acid). Remove the rubber tubes from the U-tube and fit the entrance tube by means of a good rubber stopper to the open end of the copper oxide tube, supporting the apparatus properly by means of clamps and ring stand. Attach about 60 cm. of rubber tubing to the other end of the U-tube. Pass a slow stream of hydrogen through the apparatus, and, when all the air is driven out, heat the copper oxide carefully so that all the moisture which is produced is driven over into the U-tube.

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* This weight should be preserved for Exp. 28. The copper oxide may be placed in a weighed porcelain boat, if desired.

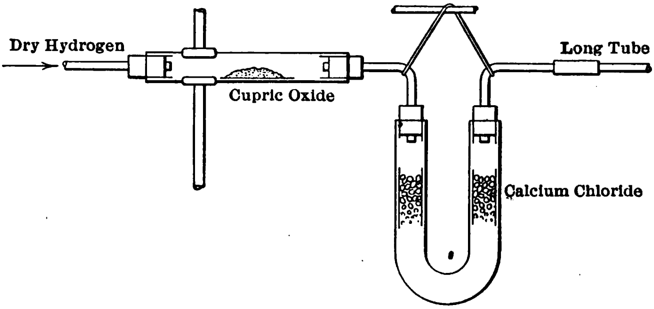

FIG. 14.

When no more moisture forms, allow the copper oxide to cool in the stream of hydrogen. When all is cool, remove the U-tube, replace the plugged stoppers, and weigh carefully. Weigh also the tube containing the reduced copper oxide.

Weight of glass tube and copper oxide...... = ... $= a$
Weight of glass tube and reduced copper.... = ... $= b$
Weight of U-tube and water.............. = ... $= c$
Weight of U-tube...................... = ... $= d$
Weight of water formed.................. = ... $= c - d$
Weight of oxygen in water................ = ... $= a - b$

$$\text{Ratio } \frac{\text{oxygen}}{\text{water}} \text{ should approximate } \frac{8}{9}.$$

COMPOUNDS AND MIXTURES

¶ 20

Experiment 27. Mix thoroughly 14 g. of iron filings with 8 g. of flowers of sulphur. Place the greater part of the mixture in a test tube. Examine the remainder under a good magnifying glass. Can you see the particles of iron and sulphur? Pass a magnet through the mass. Is the iron united chemically with the sulphur? Now heat the end of the test tube until the contents begin to glow. Remove the tube from the flame and notice how the glowing passes through the substance. Break the cooled tube and examine the contents by means of a magnet. Crush a little of the substance, and add to it a little dilute hydrochloric acid in a test tube. Has the gas which is produced any odor? Treat a little of the original mixture with dilute hydrochloric acid. Is the same gas given off? Define a mechanical mixture; a chemical compound.

EXPLANATION OF THE CONSTANT COMPOSITION OF COMPOUNDS. ATOMIC THEORY

Experiment 28. Law of Definite Proportions. ¶ 21. Prepare a copper oxide tube like that under Exp. 26, but first weigh the tube, empty, to .01 g. and then again after the copper oxide has been added. Reduce the copper oxide as before, using dry hydrogen. The calcium chloride tube is not necessary in this experiment. Instead, attach a long rubber tube to the exit tube of the apparatus to prevent the hydrogen from being ignited by the flame. Allow the reduced copper to cool in the stream of hydrogen, and then weigh the cold tube to .01 g.

Data from Exp. 26:
Weight of glass tube and copper oxide...... = ... $= a$
Weight of glass tube and copper........... = ... $= b$
Weight of glass tube....................... = ... $= e$
Weight of copper oxide.................... = ... $= a-e$
Weight of copper.......................... = ... $= b-e$

Data from Exp. 28:

Weight of glass tube and copper oxide. . . . . . = ... $=a'$
Weight of glass tube and copper. . . . . = ... $=b'$
Weight of glass tube. . . . . = ... $=e'$
Weight of copper oxide. . . . . = ... $=a'-e'$
Weight of copper. . . . . = ... $=b'-e'$

If the experiments have been carefully performed, the following proportion will be found to hold true:

$$a-e : a'-e' :: b-e : b'-e'.$$

State the Law of Definite Proportions.

Experiment 29. Law of Multiple Proportions. ¶ 21. (*a*) Repeat Exp. 28, using dry cuprous oxide instead of black cupric oxide.

Data:

Weight of glass tube and cuprous oxide. . . . . = ... $=a''$
Weight of glass tube and copper. . . . . = ... $=b''$
Weight of glass tube. . . . . = ... $=e''$
Weight of cuprous oxide. . . . . = ... $=a''-e''$
Weight of copper. . . . . = ... $=b''-e''$

Then $(a''-e'')-(b''-e'')=$ weight of the oxygen which was combined with $b''-e''$ g. of copper in the compound cuprous oxide. Similarly, in Exp. 28, $(a'-b')-(b'-e')$ is the weight of oxygen which was combined with $b'-e'$ g. of copper in the black copper oxide.

Then: $b'-e' : b''-e'' :: (a'-e')-(b'-e') : x$, where x is the weight of the oxygen which would combine with $b''-e''$ g. of copper to form black copper oxide.

$$\frac{x}{(a''-e'')-(b''-e'')}=?$$

(*b*) Just as copper unites with oxygen to form two different oxides of copper, so nitrogen unites with oxygen to form five different oxides. The parts by weight of nitrogen and oxygen in these compounds are:

	N	O
1.	63.6	36.4
2.	46.6	53.4
3.	36.8	63.2
4.	30.4	69.6
5.	25.9	74.1

Calculate the parts of oxygen which in these compounds are in combination with the *same* number of parts of nitrogen. Can the ratios existing between the numbers you have calculated be expressed by simple fractions like $\frac{1}{2}$, $\frac{1}{3}$, etc.?

(*c*) State the Law of Multiple Proportions.

(*d*) State briefly the atomic theory and show how its assumptions are in accord with the Laws of Definite and Multiple Proportions.

CHEMICAL SYMBOLS

¶ 23

Write the chemical formulæ for the following:

1. One molecule of a substance containing one atom of zinc, one atom of sulphur, and four atoms of oxygen.

2. One molecule of a substance containing two atoms of potassium, two atoms of chromium and seven atoms of oxygen.

3. Two molecules of a substance containing three atoms of calcium, two atoms of phosphorus, and eight of oxygen.*

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* Whenever a molecule contains a group of atoms which are so closely related that they often pass unchanged from molecule to molecule, the group is indicated in the formula by enclosing it in parentheses when it occurs more than once, and the number of such groups is indicated by subscripts below the right-hand parentheses, thus: CaN_2O_6 is written $Ca(NO_3)_2$.

4. Write the chemical equations for all chemical changes that occur in the experiments you have thus far performed.

Hereafter write the equations for all chemical changes that occur in your experiments.

STOICHIOMETRICAL CALCULATIONS

¶ 24

Solve the following problems:

1. How many grams of oxygen may be obtained from 500 g. mercuric oxide? What volume would the gas occupy at 20°, 700 mm.?
2. How many grams of potassium chlorate would be necessary to yield 20 g. of oxygen on heating?
3. How many kilograms of zinc would it be necessary to use in order to fill a balloon with 100 cubic meters of hydrogen at 25° and 750 mm.?
4. How many kilograms of sulphuric acid would be required in the last problem?

CHLORINE

¶¶ 25–27

Perform all experiments with chlorine under a hood.

Experiment 30. Preparation of Chlorine. (*a*) Place about 50 g. of commercial manganese dioxide (lumps) in a 500-c.c. round-bottomed flask fitted with a cork carrying an exit tube. Add enough concentrated hydrochloric acid to cover the lumps of oxide. Connect the exit tube with two Woulff bottles, the first containing water and the second concentrated sulphuric acid. Support the flask on a ring stand over a wire gauze, and heat it carefully until chlorine is freely liberated, and collect the gas in several cylinders by displacement of air, using as a delivery tube a glass tube bent at a right angle so as to give an arm about 20 cm. long.

(*b*) Mix in a test tube small quantities of salt and powdered manganese dioxide. Add a little concentrated sulphuric acid and warm the whole. Is chlorine given off?

(*c*) Heat a little powdered potassium bichromate in a test tube with concentrated hydrochloric acid.

Experiment 31. Physical Properties of Chlorine. ¶ 26. State the physical properties of the substance. Pass a stream of chlorine through a 100-c.c. flask containing cold water. If chlorine dissolves in water, preserve the solution.

Experiment 32. Chemical Properties of Chlorine. ¶27. (*a*) Have ready a hydrogen generator. Connect the exit tube with an upturned glass jet, made from a glass tube in the form of a J. When all air has been expelled * from the generator, ignite the hydrogen, and introduce the burning jet into a jar of chlorine. Notice that the color of the chlorine disappears. When the color is gone, remove the jet, and blow the breath across the top of the jar. Has a new gas formed?

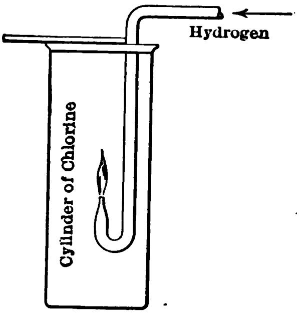

FIG. 15.

(*b*) (Two students working together.) Fit a clean 200-c.c. flask with a good cork. Find the volume of the flask and make a mark to indicate the point at which the flask would be half full. Collect the flask half full of chlorine over warm water. Complete the filling with hydrogen, keeping the light away from the mixed gases by means of a towel. Insert the cork and wire it on by means of an iron wire. Wrap a towel around the flask and tie a string about 6 meters long to one end of the towel, and carry the whole out-doors into the sunlight. Step aside and pull the towel away from the flask by means of a string. What is formed?

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* See foot-note under Exp. 18, *e*.

(*c*) Drop a pinch of finely powdered antimony into a jar of chlorine.

(*d*) Introduce a small piece of sodium into a glass tube of about 1 cm. diameter, connected with a chlorine generator, and a drying bottle with sulphuric acid. While the gas is passing, heat the tube until the sodium ignites. When the tube is cold, examine the white substance that has been formed. Moisten the finger, touch it to the white substance, (**Caution—Beware of uncombined sodium.**) and taste it. What is the product of the reaction?

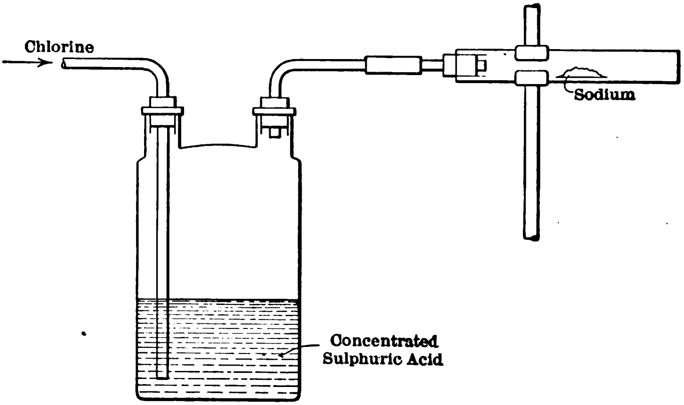

FIG. 16.

(*e*) Drop a piece of gold leaf into a test tube containing water. Pass a stream of chlorine through the water.

(*f*) Lower a little burning phosphorus in a deflagrating spoon into a jar of chlorine.

(*g*) Procure a large cylinder, fill it with chlorine and plunge a strip of filter paper saturated with hot turpentine into the gas. Explain the reaction.

(*h*) Lower a burning candle into a jar of chlorine.

(*i*) Pass chlorine gas into a beaker of hydrogen sulphide water. What is the milkiness that is formed?

(*j*) Add chlorine water to a fuchsine solution in a test tube. Do the same with indigo solution. Drop a strip of moist litmus paper into a jar of chlorine.

HYDROGEN CHLORIDE

¶ 28

Experiment 33. (*a*) **Ordinary Laboratory Method for the Preparation of Hydrogen Chloride.** Place about 25 g. of common salt in a 250-c.c. flask fitted with an exit tube and a thistle tube. Add about 50 c.c. of sulphuric acid (1: 3), warm gently, and collect several jars of the gas by displacement of air.

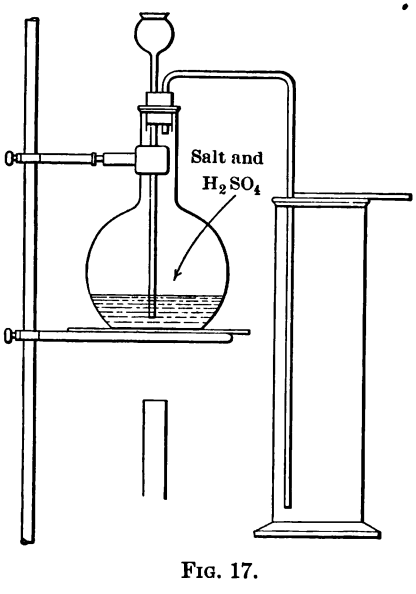

FIG. 17.

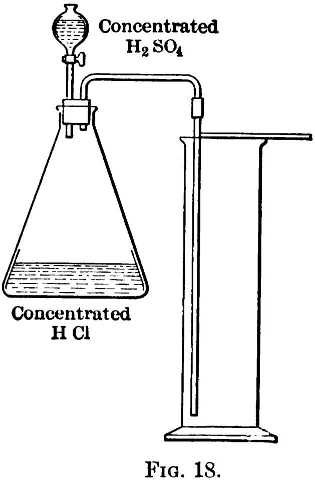

FIG. 18.

(*b*) **Methods the same in Principle as that in** (*a*). In test tubes, try the effect of concentrated sulphuric acid upon copper chloride, potassium chloride and ammonium chloride.

(*c*) **A useful Method for obtaining Hydrogen Chloride easily.** Fit a 250-c.c. flask with an exit tube and a dropping funnel. Place about 50 c.c. of concentrated hydrochloric acid in the flask and allow concentrated sulphuric acid to enter drop by drop from the funnel. Collect the gas by displacement of air. See Fig. 18.

Experiment 34. Physical Properties of Hydrogen Chloride. ¶ 29.

(*a*) State the physical properties of the gas.

(*b*) Blow the breath across the mouth of a jar of the gas.

(*c*) Open a jar of the gas under water. What is the solution called? From the manner in which the gas dissolves in water, would you be safe in assuming that it follows the Law of Henry?

Experiment 35. Some Chemical Properties of Hydrochloric Acid. ¶30.

(*a*) Test hydrochloric acid with blue litmus paper.

(*b*) Add one drop of concentrated acid to a test tube of water, mix it well, and place a drop of the dilute solution upon the tongue.

(*c*) To a piece of zinc in a test tube, add a few c.c. of dilute hydrochloric acid. What gas is given off? Do the same with some small pieces of iron. Filter, evaporate the solutions on watch-glasses, and observe the solid substances obtained. Define the terms "acid," "salt."

Experiment 36. Bases and their Relation to Acids. ¶ 30. Drop several small pieces of clean sodium about the size of a pea into a beaker of water. What is given off? Test the solution with red litmus paper. Rub a little of the solution between the fingers. Taste a minute quantity of the solution. Add one drop of phenolphthalein to the solution. Now add dilute hydrochloric acid to the solution drop by drop until the color disappears. Taste the solution. What substance does it contain? Define the term "base." What relation exists between acids and bases?

Experiment 37. Other Methods for Making Salts. ¶ **30.** (*a*) Dissolve about 1 g. of zinc oxide in sulphuric acid (1 : 5), and evaporate the clear solution to a small volume on the water-bath. Set the solution aside to crystallize. What is formed?

(*b*) To a dilute solution of silver nitrate, add a little dilute hydrochloric acid. Similarly add a little dilute sodium hydroxide to a solution of silver nitrate. What acids and bases enter into these reactions? Now add a dilute solution of sodium chloride to a solution of silver nitrate. What new salts are formed in the reaction?

COMPOSITION OF HYDROGEN CHLORIDE

Law of Gay-Lussac. ¶ 31

Experiment 38. Rub a small quantity of vaseline upon the ground edge of a *dry* glass cylinder. Collect the cylinder full of hydrogen chloride, drying the gas by passing it through concentrated sulphuric acid in a Woulff bottle. Have ready about 2 g. of powdered sodium amalgam. Cover the cylinder with a ground glass square. Open the cylinder by sliding the glass square to one side and quickly pour the amalgam into the hydrogen chloride, closing the cylinder immediately. Shake the cylinder for a few minutes, and then open it under water. Make the height of the water the same inside as outside the cylinder. Why? Note what fraction of the gas remains. Test the remaining gas for hydrogen. What is the composition of hydrogen chloride by volume? State the Law of Gay-Lussac.

When the experiment is completed, place the mercury in the special bottle kept for the purpose. *The mercury must not be thrown into the sink.*

U OF M

Experiment 39. Weight of a Liter of Hydrogen Chloride. Density of Hydrogen Chloride. Find by experiment the weight of a liter of dry hydrogen chloride and calculate the weight at 0° and 760 mm.

Calculation of the density of hydrogen chloride:

Weight of a liter of hydrogen at 0°, 760 mm.. . . . . . . $a =$ . . . . .
Weight of a liter of hydrogen chloride at 0°, 760 mm. $b =$ . . . . .
Density of hydrogen chloride $= b/a$.

Examples. 1. 1 l. of carbon dioxide at 0°, 760 mm. weighs 1.965 g. 1 l. of hydrogen at 0°, 760 mm. weighs .0895 g. Calculate the molecular weight of carbon dioxide.

2. 100 g. of carbon dioxide contain 27.27 g. carbon (i.e., CO_2 contains 27.27 per cent C.). How many grams of carbon would a gram molecule of carbon dioxide contain?

3. If no compound of carbon contains in one gram molecule less than the amount of carbon found in Ex. 2, what is the atomic weight of carbon?

4. Taking the atomic weight of oxygen as 16, calculate from the data in the previous examples the atomic weight of hydrogen and the atomic weight of carbon. Why are the atomic weights calculated from that of oxygen as 16 rather than from that of hydrogen as 1?

OZONE

¶¶ 36, 37

Experiment 40. Preparation. Obtain from your instructor an apparatus for the production of ozone, an electric cell, and an induction coil. Connect the apparatus in such a way with a tube carrying a stream of oxygen, dried by passing through concentrated sulphuric acid, that the oxygen enters at *d* (see Fig. 19). Allow the oxygen to pass slowly and connect *a* with one pole of the coil and *b* with the other pole.

MToU

Experiment 41. Properties of Ozone. Has the gas any odor? Clean a silver coin, hold it with the forceps above the flame until it is quite warm. Then let the stream of ozonized air impinge upon it. What is the bluish spot which forms on the coin? Hold a moist piece of freshly made starch potassium iodide paper[*] in the gas. Explain.

Collect some of the ozonized air in a jar. Pour a few c.c. of dilute litmus solution into the jar and shake it.

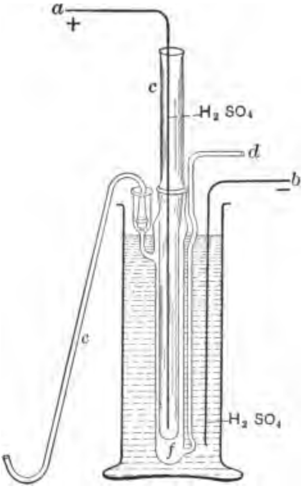

FIG. 19.

Experiment 42. Other Methods for Preparing Ozonized Air. (*a*) Place a stick of clean phosphorus in a cylinder so that it is partly covered with water.[†] Do you notice the odor of ozone in the cylinder? Hang a strip of starch-potassium iodide paper in the cylinder. Is the paper changed after half an hour?

(*b*) Under a hood place about 1 g. of finely powdered potassium permanganate in each of three 200-c.c. beakers. Moisten the permanganate with a few drops of water. Add 2 c.c. of concentrated sulphuric acid to one of the beakers, and, when a gas begins to be given off, add a pinch of flowers of sulphur. What causes the sulphur to ignite?

Using a second beaker, ignite phosphorus in the same way.

Allow a stream of illuminating gas (issuing from a long glass tube drawn to a small opening) to impinge upon the surface of the decomposing permanganate in a third beaker until ignition of the gas takes place.

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[*] Make a few c.c. starch solution (see Exp. 54), add a few c.c. potassium iodide solution, and moisten strips of filter paper with the mixture.

[†] If the class is large, the instructor should prepare a single cylinder with several sticks of phosphorus partly covered with water. The students may then test the air in the cylinder for ozone.

(*c*) Test the oxygen issuing from a Hofmann's apparatus for ozone.

HYDROGEN PEROXIDE

¶¶ 38, 39

Experiment 43. Preparation of Hydrogen Peroxide. (*a*) To 50 c.c. of cold dilute sulphuric acid in a 100-c.c. beaker, add with constant stirring about 10 g. of finely powdered barium peroxide. After the precipitate has settled, decant the clear solution through a filter. What does the filtrate contain? Preserve for *c*, 5.

(*b*) **Instability of Hydrogen Peroxide.** Pour a little hydrogen peroxide solution into a test tube containing a little powdered manganese dioxide.

(*c*) **Oxidizing Action.** 1. To a test tube containing one drop of dilute lead nitrate, add 4 c.c. of water, and pass a little hydrogen sulphide through the solution. The black precipitate is lead sulphide. Now add 5 c.c. of hydrogen peroxide, shake the contents of the tube, and allow it to stand. Explain the change from black to white.

2. To a solution of barium hydroxide in a test tube, add hydrogen peroxide solution. What is the white precipitate formed?

3. To a sulphuric acid solution of titanium dioxide, add hydrogen peroxide. To what is the yellow color due?

4. Test hydrogen peroxide solution with potassium iodide-starch paste solution in the presence of a little ferrous sulphate. Explain.

5. To a very dilute solution of potassium bichromate add a little dilute sulphuric acid, and then a little hydrogen peroxide. Add about 3 c.c. of ether and shake the tube, closing the opening

with the thumb. The blue ether solution is a test for hydrogen peroxide.

(*d*) **Reducing Action.** 1. To a solution of silver nitrate add a very little dilute sodium hydroxide, and then a little hydrogen peroxide. Explain.

2. Add hydrogen peroxide to a not too dilute solution of potassium permanganate acidulated with sulphuric acid. Does any gas escape? What is it? Explain this and the change of color.

3. To a very little peroxide of lead in a test tube, add some hydrogen peroxide. Explain.

OSMOTIC PRESSURE

Problems. 1. In Pfeffer's experiments upon the osmotic pressure of sugar solutions, he found that a 1 per cent solution at 7° gave a pressure of 535 mm. of mercury. Assuming this to be correct for a 1 per cent solution, calculate the pressure for a 6 per cent solution.

2. If a 1 per cent solution of sugar gives a pressure of 535 mm. at 7°, calculate the pressure at 80°.

3. If a 1 per cent solution of sugar gives a pressure of 535 mm. at 7° calculate the pressure of a 5 per cent solution at 100°.

4. What would the gaseous pressure of the sugar in a 1 per cent solution be at 7°, if the sugar could be volatilized without decomposition and be enclosed in a volume equal to that of the solution, at the same temperature?

MOLECULAR WEIGHT BY MEANS OF THE LOWERING OF THE FREEZING POINT

¶¶ 40-43

Experiment 44. Fit a test tube 15 cm. long and 4 cm. in diameter with a cork carrying a thermometer giving readings between 50° and 100° and graduated to .1°. Let there be a small hole fitted with a short glass tube in the cork, through which a stirrer may move up and down. The stirrer is made of a glass rod bent at its lower end in a circle of such a size that

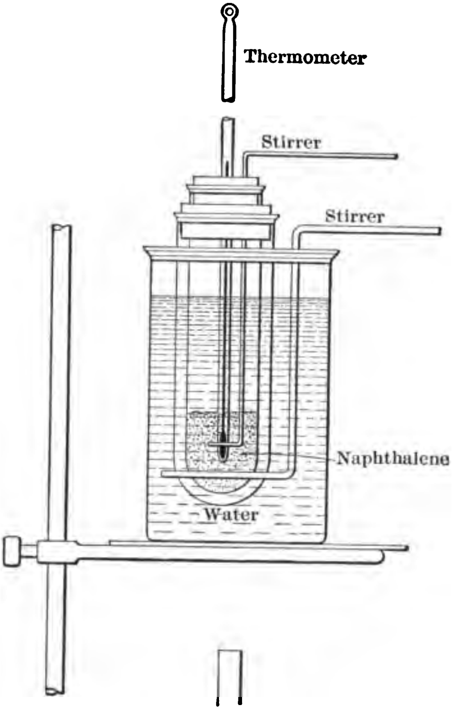

FIG. 20.

it may be moved freely in the tube. Fit the test tube by means of a cork into a larger test tube.

Weigh out into the tube 40 g. pure naphthalene. Support the whole in a beaker of water, containing a stirrer larger but similar to the one described above, so that the greater portion of the tube is submerged. Heat the water until all the napthalene is melted. Then allow the water to cool, and when the temperature reaches the neighborhood of 80° stir both the water and the naphthalene. When crystals of naphthalene begin to form, note the temperature when it remains constant. This is the freezing point of naphthalene.

Now, dissolve about 3 g. of dichlorbenzene weighed to .01 g., in the naphthalene, and repeat the experiment.

Weight of naphthalene........................ = ... $= a$
Weight of dichlorbenzene.................... = ... $= b$
Freezing point of naphthalene................ = ... $= c$
Freezing point of solution.................... = ... $= d$
Molecular lowering of naphthalene........... = 71

The percentage strength of the solution will be $\frac{b \times 100}{a}$.

The lowering of a 1 per cent solution will then be $\frac{a(c-d)}{100b}$.

Therefore M,

the molecular weight of dichlorbenzene will be $\frac{71 \times 100b}{a(c-d)}$.

Your result should approximate 147.

BROMINE

¶ 44

Experiment 45. Preparation. (*a*) To a test tube containing a solution of potassium bromide, add a little chlorine water. What is set free?

(*b*) Place 5 g. of powdered potassium bromide and 5 g. of powdered manganese dioxide in a retort, and mix them by shaking the retort. Add 50 c.c. of dilute sulphuric acid, and distil, catching the distillate in a receiver kept cool by being immersed in cold water, or by having cold water running over it, as indicated in Fig. 21.

Experiment 46. Physical Properties. (*a*) Place a drop of bromine in a cylinder. **Caution.** *Great care must be taken in handling bromine. A drop on the skin produces a severe wound. Do not breathe bromine fumes.* Is bromine volatile?

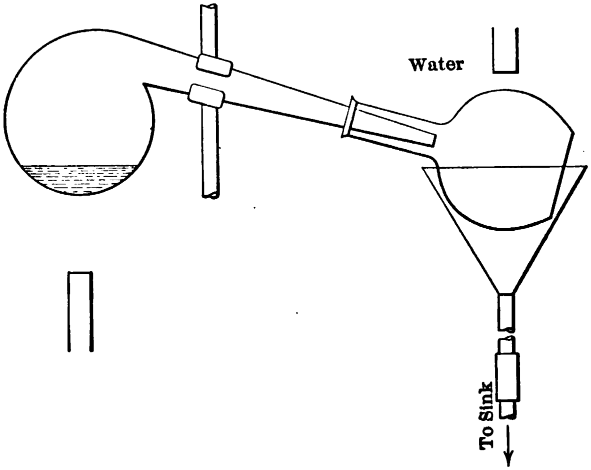

Fig. 21.

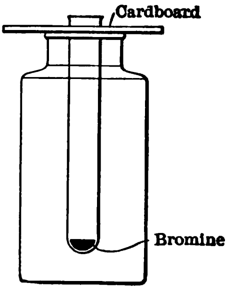

Fig. 22.

(*b*) Place a drop of bromine in a 250-c.c. flask, and fill the flask half full of water. Cork and shake the flask. Is bromine soluble in water?

(*c*) Place 5 c.c. of bromine water in each of three test tubes. Add 1 c.c. of ether, benzene, and carbon bisulphide to the test tubes respectively, and shake them well. What can you say about the solubility of bromine?

Experiment 47. Chemical Properties of Bromine. (*a*) Burn hydrogen in a jar of bromine vapor, just as you burned hydrogen in chlorine. Test the product as in Exp. 32, *a*. What is formed?

(*b*) Cut a hole large enough to hold a test tube in a piece of pasteboard. Place the tube in a large-mouthed bottle so that the pasteboard supports the tube. See Fig. 22. Set the whole under a hood, add a few drops of bromine (not bromine water). Drop a 3-mm. piece of potassium into the tube by means of forceps. Explain.

(*c*) Try a similar experiment, using sodium instead of potassium.

(*d*) Perform a similar experiment, using powdered antimony instead of potassium.

HYDROGEN BROMIDE

¶ 45

Experiment 48. (*a*) **Preparation from the Elements.** Have ready a hydrogen generator. Fit a test tube with a cork containing an entrance tube extending almost to the bottom and a short exit tube. Connect the test tube with the generator and with a bulb tube containing platinized asbestos and fitted with an exit tube bent upward and drawn out to a small open-

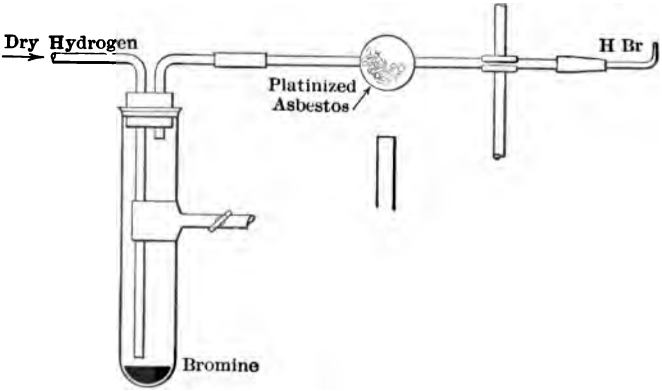

FIG. 23.

ing. Heat the bulb tube and pass a mixture of hydrogen and bromine vapor through the apparatus. Test the issuing gas by means of a piece of moist blue litmus paper, and by blowing breath across the stream of gas. What compound has been formed? Perform the experiment under a hood.

(*b*) **Preparation from a Bromide by Reduction.** Reduce a little lead bromide in a reduction tube by means of hydrogen and test the gas. See Exp. 29 for the apparatus.

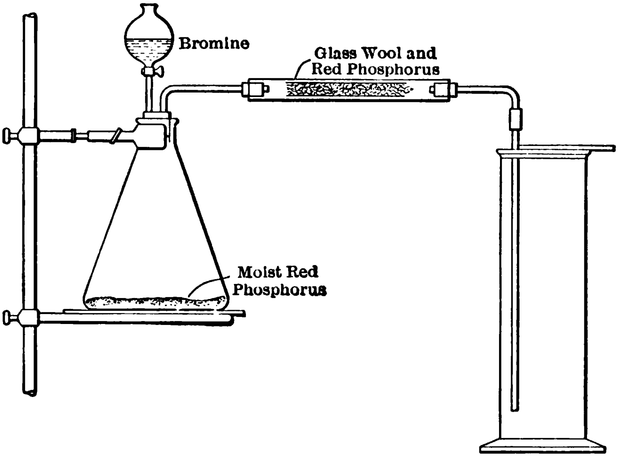

FIG. 24.

(*c*) **Preparation from Naphthalene and Bromine.** Allow bromine to drop from a dropping funnel into pure naphthalene in a 200-c.c. flask, and test the gas which is evolved.

Experiment 49. Preparation from Phosphorus Pentabromide. One of the Most Convenient Methods for Obtaining a Stream of Hydrogen Bromide Gas. Place about 5 g. of red phosphorus in a 250-c.c. flask and add about 5 c.c. of water. Fit the flask with a two-holed stopper, carrying an exit tube and a dropping funnel. Connect the flask with a "20-cm.

glass " tube containing glass wool impregnated with red phosphorus, and fitted with an exit tube as in Fig. 24. Allow bromine to drop upon the wet phosphorus and collect the hydrogen bromide by displacement of air in cylinders. Why is the tube with phosphorus necessary? Explain the various steps of the reaction.

Experiment 50. Properties of Hydrogen Bromide. Blow the breath across the open mouth of a jar of the gas. Plunge some moist blue litmus paper into the gas. Compare the odor of the gas with that of hydrogen chloride. Open a jar of the gas under water. Taste a dilute solution of the gas.

Try the effect of hydrobromic acid solution upon magnesium.

To a little potassium bromide in a test tube, add a little concentrated sulphuric acid. Is hydrogen bromide given off? Is any other substance formed?

Experiment 51. Tests for Hydrobromic Acid and Bromides. (*a*) To a solution of hydrobromic acid, in a test tube, add a little dilute silver nitrate.

(*b*) Pass hydrogen sulphide into bromine water until the water is decolorized. Boil the solution ten minutes to free from hydrogen sulphide, filter, and test it with silver nitrate. What has been formed?

IODINE

¶¶ 46, 47

Experiment 52. (*a*) **Preparation of Iodine by the Oxidation of Hydrogen Iodide.** Heat gently a mixture of 2 g. powdered potassium iodide, 4 g. powdered manganese dioxide, and 15 c.c. concentrated sulphuric acid in a small beaker, keeping the beaker covered with a porcelain dish containing cold water. Examine the iodine which collects on the bottom of the dish.

(*b*) **Preparation of Iodine by the Replacing Action of Chlorine.** To a solution of potassium iodide in a test tube, add a little chlorine water. Add 3 c.c. of ether, shake the tube, and draw off the ether with a pipette. Allow the ether to evaporate spontaneously, and examine the residue.

Experiment 53. Properties of Iodine. Examine the substance, and describe its appearance. Place a crystal in a 200-c.c. flask, and heat it gently. Examine the upper part of a bottle that has contained iodine for some time. What can you say of the volatility of iodine? Try the solubility of iodine in water, a solution of potassium iodine, ether, chloroform, benzene, alcohol, and carbon disulphide, using a very small crystal of iodine in each case.

Grind a drop of mercury with a little iodine in a mortar.

Under a hood, heat a little arsenic and iodine together in a test tube.

Experiment 54. Test for Iodine. Make an emulsion of starch paste as follows: Grind a piece of starch in a mortar with some cold water. Heat 50 c.c. of water in a beaker to boiling, and add the starch. This is commonly called "starch solution," though it is not a true solution. To a very dilute solution of iodine in water or in potassium iodide solution, add a little starch solution. The brilliant blue color formed is the most delicate test for free iodine. Heat the solution to boiling until the color disappears. Allow the solution to cool and observe the return of the blue color.

HYDROGEN IODIDE

¶ 48

Experiment 55. Best Method for Preparing Hydrogen Iodide.* Place 22 g. iodine in a 200 c.c. round-bottomed flask, and add 2 g. of dry phosphorus cut into small pieces, adding the pieces one at a time. When the phosphorus triiodide is cool, add 3 c.c. of water, and connect the flask immediately with a calcium chloride tube containing glass wool and red phosphorus as in Exp. 49. Collect the gas in cylinders by displacement of air.

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* On account of the cost of iodine, several students should perform this experiment together.

Experiment 56. Properties of Hydrogen Iodide. (*a*) Compare hydrogen iodide with hydrogen bromide and hydrogen chloride as to color, fuming in air, acidity, taste of solution, etc. Open a jar of the gas under water. Hold a hot glass rod in a jar of hydrogen iodide. Pour 1 c.c. of hot fuming nitric acid into a jar of hydrogen iodide. Examine a solution of hydrogen iodide, which has been exposed to the light for some time.

(*b*) **Tests.** Test solutions of hydrogen iodide and of potassium iodide with dilute silver nitrate and with lead nitrate solutions.

(*c*) To a test tube containing a few crystals of potassium iodide, add a little concentrated sulphuric acid. Can this be used as a method for the preparation of hydrogen iodide? Explain.

DISSOCIATION

¶¶ 49–51

Examples. 1. How many grams are there in a mole of hydrogen iodide? of hydrogen? of iodine?

2. St. Claire Deville found that hydrogen iodide at 445° yielded 79 per cent of unchanged gas and 21 per cent of a mixture of hydrogen and iodine. Starting with one gram molecule of hydrogen, calculate the concentration equilibrium constant.

3. From the data in the preceding example, assuming the pressure to be one atmosphere, calculate the partial pressures of hydrogen iodide, hydrogen, and iodine respectively. Calculate the pressure equilibrium constant.

In the case of the dissociation of hydrogen iodide, the gas volume does not change, since two molecules of hydrogen iodide (2HI) yield two molecules (H_2 and I_2). When the volume changes, as in the case of

$$PCl_5 \rightleftarrows PCl_3 + Cl_2,$$

the volume must be taken into consideration.

Example. Phosphorus pentachloride at 250° and one atmosphere is .8 dissociated. Starting with one gram molecule, calculate the concentration equilibrium constant.

Since one gram molecule occupies 22.4 l. at 0° and 760 mm. pressure, this would occupy, if undissociated, at 250° and 760 mm.

$$22.4\left(\frac{273+250}{273}\right)\text{l.}$$

Since .8 is dissociated, the volume has increased $1-.8+2\times.8$ or 1.8 times the volume when undissociated. Therefore the volume of the dissociated gas at 250° is

$$1.8\times22.4\left(\frac{273+250}{273}\right)\text{l.}$$

Since the concentration is the number of gram molecules in 1 l., the concentration of the undissociated phosphorus pentachloride is

$$\frac{.2}{1.8\times22.4\left(\frac{273+250}{273}\right)}.$$

and the concentration of the phosphorus trichloride and the chlorine each is

$$\frac{.8}{1.8\times22.4\,\frac{273+250}{273}}.$$

Therefore, since the concentration equilibrium constant is

$$\frac{\text{Conc. } PCl_3\times\text{Conc. } Cl_2,}{\text{Conc. } PCl_5}$$

we have

$$\frac{.8^2}{.2\times1.8\times22.4\,\frac{273+250}{273}}.$$

4. Carbon dioxide at 3000° and one atmosphere pressure is .4 dissociated into carbon monoxide and oxygen according to the reaction

$$2CO_2\leftrightarrows2CO+O_2.$$

Calculate the concentration equilibrium constant.

FLUORINE

¶ 52

Experiment 57. (*a*) Draw a diagram of Moissan's apparatus for preparing fluorine.

(*b*) State the physical and chemical properties of fluorine as Moissan found them.

HYDROGEN FLUORIDE

¶ 53

Experiment 58. (*a*) **Preparation of Hydrogen Fluoride.** In a shallow lead or iron dish, place about 20 g. of powdered calcium fluoride (fluor spar). Add enough concentrated sulphuric acid to make a thin paste. On a glass plate, place a small quantity of paraffine. Warm the plate so that the paraffine on melting forms a *thin* layer completely covering the surface of the plate. By means of a needle or pin, sketch a diagram of some object on the plate, and place it upon the dish so that the diagram is exposed to the vapors of hydrogen fluoride which are given off. Place the whole under a hood, and examine it the next day.

(*b*) **Properties of Hydrogen Fluoride.** 1. In a test tube, place a little calcium fluoride, and add about 1 c.c. of concentrated sulphuric acid. Heat the tube under a hood and test the escaping gas with moist, blue litmus paper. Do not inhale any of the gas. Empty the tube, and rinse it with water. Has the tube been attacked?

2. Moisten the exposed lines on a plate similar to the one in (*a*) with hydrofluoric acid. After a few minutes, clean the plate and compare the etching with that obtained in (*a*).

3. Try the effect of dilute hydrofluoric acid on a piece of zinc.

CHLORINE DIOXIDE

¶ 57

Experiment 59. Preparation and Properties of Chlorine Dioxide. Place about 5 c.c. of concentrated sulphuric acid in a cylinder about 20 cm. high. Sprinkle powdered potassium chlorate into the acid under a hood. When the jar is full of a yellow gas, plunge a hot glass rod into it. The gas may decompose with a detonation or simply decolorize. How does the odor of the gas differ from that of chlorine?

Experiment 60. (*a*) **Oxidizing Power of Chlorine Dioxide.** Mix gently with a feather on a piece of paper about 1 g. of powdered sugar with an equal amount of powdered potassium chlorate. Place the mixture upon a piece of asbestos under the hood, and by means of a long glass tube, allow a drop of concentrated sulphuric acid to fall upon the mixture. Explain the reaction.

(*b*) Hang a test tube in a wide-mouthed bottle as in Exp. 47 (*b*). Place about 2 g. potassium chlorate in the tube, add water until the tube is about two-thirds full, add a small piece of yellow phosphorus, and by means of a glass tube allow a few drops of sulphuric acid to flow directly upon the phosphorus and chlorate.

Experiment 61. Solution of Chlorine Dioxide. Place a porcelain crucible in an evaporating dish about 10 cm. in diameter containing a layer of water about 2 cm. deep. In the crucible, place 2 g. potassium chlorate, and add a cooled mixture of 8 c.c. sulphuric acid and 2 c.c. water. Cover the whole with a glass plate. What does the yellow solution thus obtained contain?

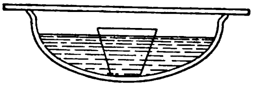

FIG. 25.

Add a solution of potassium hydroxide to the above solution until it no longer changes a piece of blue litmus paper red. What is formed in the solution?

CHLORIC AND PERCHLORIC ACIDS

¶¶ 59, 60

Experiment 62. Properties of Chloric Acid. ¶ 59. (*a*) Add a little chloric acid to a solution of indigo, then add a little sulphurous acid.

(*b*) To a test tube containing a little chloric acid, add a little concentrated hydrochloric acid, and warm the mixture. Is chlorine given off?

(*c*) Pass hydrogen sulphide through a dilute solution of chloric acid. What is precipitated?

Experiment 63. Perchloric Acid. ¶ 60. (*a*) Try the effect of perchloric acid upon hydrochloric acid and upon sulphurous acid.

(*b*) To a very strong solution of potassium chloride, add perchloric acid or sodium perchlorate solution. What are the crystals which are formed?

OXYGEN COMPOUNDS OF IODINE

¶¶ 62–66

Experiment 64. Iodic Acid. (*a*) To 1 g. of powdered iodine in a porcelain dish, add 10 c.c. of concentrated nitric acid and cover the dish with a watch-glass. Warm the dish, and add 1 c.c. concentrated nitric acid from time to time, until the brown color disappears. Evaporate the residue on the water-bath. What are the crystals?

(*b*) Dissolve about 1 g. iodic acid in a little water, add a little concentrated hydrochloric acid, and warm the mixture. Do you detect an odor of chlorine?

(*c*) To a similar solution of iodic acid, add a few drops of hydriodic acid which has been carefully clarified, if brown, by means of sodium thiosulphate. What is set free?

ELECTROLYTIC DISSOCIATION

¶ 65

Examples. 1. The experimentally determined osmotic pressure of a solution of ionized salt which ionizes into two ions is 684 mm. The osmotic pressure as calculated from the molecular weight is 360 mm. Find the degree of dissociation.

2. The molecular conductivity of the above solution is 81. At a dilution so great that addition of more water does not affect the molecular conductivity, the value is 90. What is the degree of dissociation?

Experiment 65. Ionic Equilibria. Colors of Ions. ¶ 66. (*a*) Make a *concentrated* solution of sodium chloride in a test tube. Add a little concentrated hydrochloric acid. What is the precipitate? Explain its formation.

(*b*) Since there are many colorless chloride, bromide, nitrate, and sulphate solutions, what must be the color of the Cl, Br, NO_3, SO_4 ions? Examine dilute solutions of cupric chloride, copper nitrate, and copper sulphate. What is the color of the Cu ion? Examine dilute solutions of ferric chloride, nickel nitrate, cobalt nitrate, potassium chromate, potassium bichromate and potassium permanganate, and determine the colors of the Fe(ic), Ni, Co, CrO_4, Cr_2O_7, MnO_4 ions.

(*c*) Make up a *very concentrated solution* of cupric bromide in a test tube. To a little of this solution in another test tube, add water gradually, noting any color changes. What is the color of the molecule, $CuBr_2$, in solution? What is the color of the Cu ion?

(*d*) Divide the diluted cupric bromide solution into two parts. To one add about 3 g. of solid potassium bromide, and to the other about 5 g. of solid cupric chloride. Explain the color changes.

Experiment 66. Acids and Bases. ¶ 66. Define " acid " in terms of the ionic theory. Similarly define " base." How do strong acids differ from weak ones? How do strong bases differ from weak ones?

(*a*) To a solution of a little sodium carbonate in a test tube, add a little hydrochloric acid. How do you explain the liberation of carbon dioxide from carbonates by means of hydrochloric acid?

(*b*) Dissolve 1g. of sodium fluoride in about 1 c.c. of water in each of two test tubes. To one add an equal volume of concentrated hydrochloric acid, close the two tubes with corks, and let them stand for about two hours. Then rinse the tubes out and examine them. Explain the difference in the appearance of the glass of the two tubes.

Experiment 67. Hydrolytic Dissociation. ¶ 66. (*a*) Test solutions of sodium carbonate and potassium cyanide with red litmus paper. Explain why the solutions are alkaline.

(*b*) Test solutions of aluminum sulphate and copper nitrate with blue litmus paper. Explain why these solutions are acid.

SULPHUR

¶¶ 67–69

Experiment 68. Physical Properties of Sulphur. (*a*) Dissolve about 2 g. of crushed sulphur in the least possible amount of carbon bisulphide in a test tube. Allow the solution to evaporate on a watch-glass, and examine with a magnifying glass the crystals which separate out.

(*b*) Fill a test tube about one-fourth full of roll sulphur. Heat the tube carefully until the sulphur is all melted. Continue heating the tube, and note at the same time what changes appear in the color and consistency of molten sulphur.

Experiment 69. Allotropic Forms of Sulphur. ¶68. (*a*) **Rhombic sulphur.** See Exp. 68 (*a*).

(*b*) **Monoclinic Sulphur.** Melt very slowly enough roll sulphur in a 100-c.c. beaker to fill it about one half. Allow the liquid to cool until crystals begin to form across the surface, then pour out the liquid sulphur that remains. Compare the crystals with those obtained in (*a*). After a few hours, examine the crystals again.

(*c*) **Amorphous Sulphur.** 1. **Plastic Variety.** Reheat the test tube of sulphur, used in Exp. 68 (*b*), until the sulphur boils. Then quickly pour out the molten sulphur in a thin stream into a dish of cold water. Describe the product. Set a piece aside and examine it again the next day. Dry a piece of plastic sulphur and treat it with carbon bisulphide in a test tube. Does all dissolve? Explain the presence of the two forms of sulphur.

2. **Milk of Sulphur.** To 20 c.c. of yellow ammonium sulphide solution in a beaker, add concentrated hydrochloric acid to excess under a hood. Allow the precipitate to settle, pour off the liquid, and set the beaker aside to dry. When it is completely dry, test the solubility of the sulphur in carbon bisulphide.

Experiment 70. Chemical Properties of Sulphur. ¶ 69. (*a*) Reread Exp. 14 (*c*).

(*b*) Into a test tube containing *boiling* sulphur, plunge a strip of copper foil by means of a pair of forceps.

(*c*) Rub a little mercury and sulphur together in a mortar.

HYDROGEN SULPHIDE

¶¶ 72, 73

Experiment 71. Preparation of Hydrogen Sulphide. To a few pieces of ferrous sulphide in a test tube, add a little dilute hydrochloric acid, and note any gas that may be given off. Obtain from your instructor a Kipp apparatus for generating hydrogen sulphide. See Fig. 26.

Experiment 72. (*a*) **Physical Properties of Hydrogen Sulphide.** Study hydrogen sulphide with respect to its color, odor, and solubility in water. To test the solubility, pass the gas slowly through a beaker of cold water for about ten minutes under a hood. Remove the beaker, blow out the excess of gas and test the liquid for odor. Boil this solution, testing the vapors from time to time with filter paper moistened with lead acetate solution.

(*b*) **Chemical Properties of Hydrogen Sulphide.** 1. Collect a cylinder of hydrogen sulphide by air displacement. Ignite the gas. What is formed? What remains on the sides of the cylinder? Did complete combustion take place?

2. Fill a cylinder a little over two-thirds full of oxygen by water displacement. Complete the filling with hydrogen

sulphide, remove the cylinder from the water, invert it several times, and ignite the gas mixture. Does complete combustion take place?

3. Examine a laboratory bottle which has contained hydrogen sulphide solution for some time. What is the deposit on the bottom of the bottle? Explain its presence.

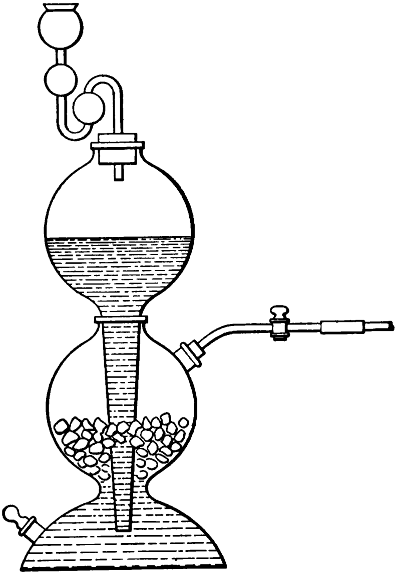

FIG. 26.

4. Reread Exp. 51 (*b*).

5. Pass hydrogen sulphide through a solution of potassium bichromate made acid with dilute sulphuric acid.

6. Pour a little hot fuming nitric acid from a test tube into a cylinder of hydrogen sulphide gas.

7. Test a solution of hydrogen sulphide with blue litmus paper.

Experiment 73. Use of Hydrogen Sulphide in Analysis. **¶ 73.** (*a*) Into dilute, slightly acid solutions of mercurous nitrate, bismuth nitrate, antimony chloride, potassium arsenite, silver nitrate, and cadmium nitrate, lead a stream of hydrogen sulphide.

(*b*) Try the effect of hydrogen sulphide upon similar solutions of ferrous chloride, aluminum nitrate, cobalt nitrate, zinc nitrate, and nickel nitrate.

(*c*) Try the action of ammonium sulphide on solutions similar to those in (*b*).

Experiment 74. Solubility Product. (*a*) **¶ 73.** Pass hydrogen sulphide through a dilute, slightly acid solution of copper sulphate for about five minutes. Filter off the precipitate, boil the filtrate for ten minutes, and test the filtrate for copper by means of ammonium hydroxide solution. Any copper left will give a blue color with ammonium hydroxide. Is copper completely precipitated by hydrogen sulphide? Explain your results by means of the electrolytic dissociation theory and the law of mass action.

(*b*) Suspend the copper sulphide precipitate obtained in (*a*) in about 100 c.c. of water. Add 2 c.c. of nitric acid. Filter and test the filtrate for copper as before. Explain why the copper sulphide is not totally dissolved.

(*c*) Pass hydrogen sulphide into a dilute acid solution of ferrous sulphate. Is any precipitate formed? Now add a slight excess of ammonium sulphide, and filter the solution. Test the filtrate for iron with potassium ferrocyanide solution, which will give a blue color, if any iron is present. Now add dilute acid to a suspension of the precipitate in 100 c.c. of water. Explain why hydrogen sulphide does not precipitate iron sulphide, why ammonium sulphide does, and why the resulting precipitate dissolves in dilute acids.

(*d*) Pass hydrogen sulphide into dilute solutions of barium chloride and calcium chloride. Why does no precipitate form?

HALOGEN COMPOUNDS OF SULPHUR

¶ 75

Experiment 75. Sulphur Monochloride. (*a*) Set up a chlorine generator, connect it with two Woulff bottles, one containing water and the second containing concentrated sulphuric acid. Fit a 100-c.c. round-bottomed flask, containing a few pieces of sulphur, with a good two-holed cork. Fit the cork with an entrance tube reaching nearly to the bottom of the flask and with an exit tube bent at an angle of 30°. Connect the exit tube with a dry, 300-c.c. flask and set the whole apparatus under a hood, connecting the flask containing the sulphur with the chlorine apparatus. Keeping the sulphur molten, pass a slow stream of chlorine through the apparatus. Describe the product formed. Test a little of it under the hood with water. What reaction takes place?

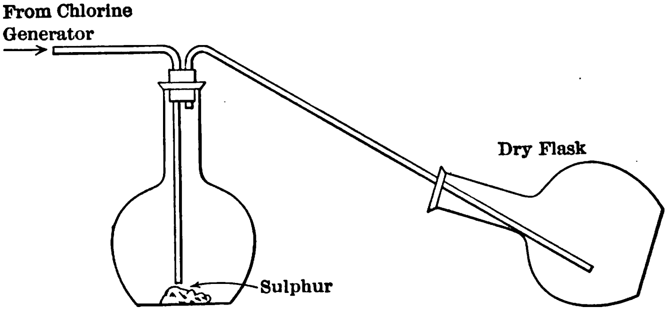

FIG. 27.

(*b*) **Sulphur Dichloride.** Examine a little sulphur dichloride. Describe the substance. How do you know that the liquid is not a mixture of chlorine and sulphur monochloride? See ¶75.

VALENCE

¶ 76

(*a*) Define valence. What is meant by the valence of an ion?

(*b*) Write the graphic formulæ of all the compounds you have studied thus far.

OXIDES OF SULPHUR

¶¶ 78–80

Experiment 76. (*a*) **Preparation of Sulphur Dioxide. ¶ 78.** 1. Place about 20 g. of copper turnings in a 500-c.c. flask. Fit the flask with a two-holed cork containing a safety tube and an exit tube. Add enough concentrated sulphuric acid to

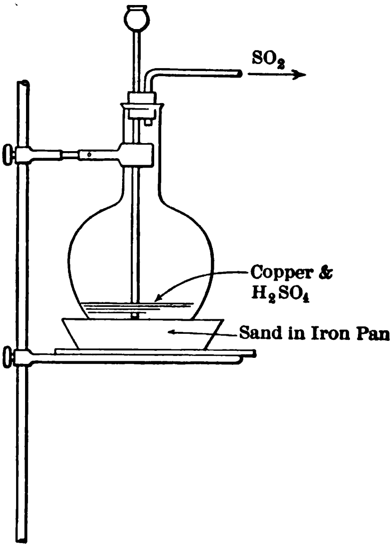

FIG. 28.

cover the copper turnings. Set the flask upon a sand bath, supporting the whole firmly upon a ring stand. Heat the flask under a hood and collect the gas in cylinders by displacement of air. Could you use carbon or sulphur instead of copper, and, if so, what difference would there be in the products?

2. Heat a little pyrite in the air by means of a blowpipe. Do you detect the odor of sulphur dioxide?

3. Heat a mixture of copper oxide and flowers of sulphur in a test tube. Is sulphur dioxide formed?

4. Mix equal parts of copper oxide and copper sulphide in a test tube and heat the mixture. Explain the formation of sulphur dioxide.

(*b*) **Properties of Sulphur Dioxide and Sulphurous Acid.** 1. What are the physical properties of sulphur dioxide, such as color, odor, etc.?

2. Add a little water to a cylinder of sulphur dioxide. Does the gas dissolve? Taste a drop of the liquid. Test the liquid with blue litmus paper. What does the solution contain?

3. Place a piece of moist red litmus paper in a cylinder of sulphur dioxide. Explain the bleaching of the paper.

4. Moisten a piece of filter paper with potassium iodate-starch solution and introduce the paper into a cylinder of sulphur dioxide.

5. Ignite a piece of magnesium ribbon and plunge it into a cylinder of sulphur dioxide.

6. Pass hydrogen sulphide and sulphur dioxide together into a cylinder or Woulff bottle under a hood. Explain the formation of sulphur and moisture. See Fig. 29.

(*c*) **Preparation of Sulphur Trioxide. ¶ 79.** Under a hood, have ready a gas-holder containing oxygen, and an apparatus for generating dry sulphur dioxide. By means of a Y tube connect the gas-holder and the sulphur dioxide generator with a Woulf bottle containing sulphuric acid. Pass the mixed gases slowly through a bulb tube containing platinized asbestos, gently heating the bulb with the flame of a Bunsen burner, and collect the product of the reaction in a test tube imbedded in a mixture of ice and salt. After about ten minutes disconnect the test tube and examine the substance which has condensed in it.

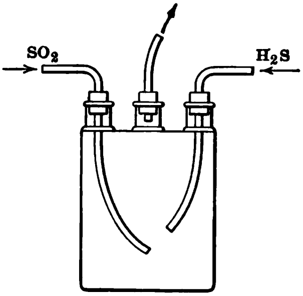

FIG. 29.

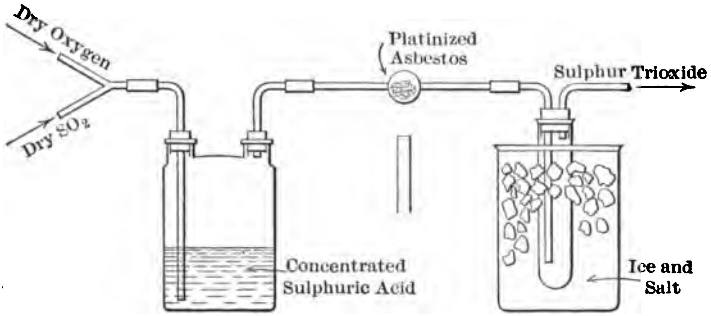

FIG. 30.

(*d*) **Properties of Sulphur Trioxide.** Give the physical properties of the substance made in (*c*). What is the effect of water upon it? Preserve the solution for use in Exp. 78 (*a*).

OXYACIDS OF SULPHUR

¶¶ 81–88

Experiment 77. (*a*) **Sulphurous Acid.** ¶ **84.** Refer to Exp. 14 (*c*) and Exp. 76 (*b*), 2, 3, concerning a solution of sulphur dioxide in water.

(*b*) **Sulphites.** To a solution of sodium sulphite, add a little dilute hydrochloric acid. Try the effect of a solution of a sulphite upon a dilute potassium permanganate solution.

Experiment 78. Preparation of Sulphuric Acid. ¶¶ **85–88.** (*a*) Test with barium chloride solution the solution obtained in Exp. 76 (*d*). Is the white precipitate thus formed soluble in dilute acids?

(*b*) In a glass tube closed at one end and about 1 cm. in diameter, place a little powdered potassium sulphate and then a small piece of solid phosphoric acid. Heat the middle part of the tube in the flame, draw it out to a diameter of about 3 mm. and bend it so that the tube becomes a small retort. When the tube is cool, support it by means of a clamp and heat the mixture, so that the sulphuric acid which forms distils over. Test the diluted distillate with barium chloride solution.

(*c*) Boil a little sulphur with concentrated nitric acid in a test tube, dilute the acid, filter and test the solution with barium chloride solution.

(*d*) Heat a small quantity of potassium nitrate in a porcelain crucible until it begins to give off oxygen. Then add a little at a time a small quantity of sulphur. After the molten mass has cooled, dissolve it in water, filter and test it with barium chloride solution.

(*e*)* Fit a wide-mouthed bottle, about 20 cm. high, with a good three-holed cork. Fit a 100-c.c. round-bottomed flask, containing about 15 c.c. concentrated nitric acid, with a two-holed cork carrying an entrance tube reaching below the surface of the acid and an exit tube bent twice at right angles so as to have an arm about 10 cm. long horizontally and 10 cm. long perpendicularly. Fit the outer end of the entrance tube with a short piece of rubber tubing and a pinch cock. In a hard glass tube about 15 cm. long, place a plug of glass wool and then a porcelain boat containing about 5 g. of sulphur. Fit the whole in a horizontal position to the large bottle by means of a cork

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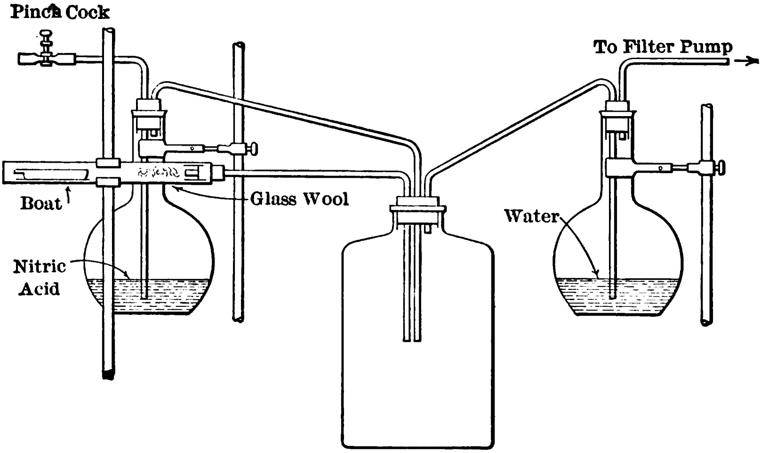
* This apparatus may be set up once for all by the instructor and used by different students successively.



and a glass tube bent at a right angle. Fit the opening of the hard glass tube with an entrance tube carrying a piece of rubber tubing with a pinch cock. Connect the nitric acid flask with the large bottle by means of the second hole of the large cork. Connect the large bottle by means of the third hole with a wash-bottle containing water, and connect the latter with a filter pump. Draw air through the apparatus slowly, heating the sulphur until it burns, and, at the same time, warm the nitric acid. Regulate the apparatus by means of the pinch cocks so that the sulphur dioxide from the burning sulphur decolorizes the red fumes before more nitric acid fumes are intruduced into the bottle. If all is done properly, the sides of the bottle will become covered with crystals (chamber crystals). Sometimes the crystals fail to appear since they are extremely soluble in water. After a sufficient amount of crystals or liquid has collected in the bottle, disconnect the tube from the nitric acid flask, draw air through the bottle, remove the cork, and add a few cubic centimeters of water. Filter the liquid and test it for sulphuric acid.

Experiment 79. Properties of Sulphuric Acid. ¶ 87. (*a*) Determine the specific gravity of concentrated sulphuric acid by means of an ordinary hydrometer, and then by means of a Baumé aerometer. Compare the meaning of the two readings.

(*b*) Dilute a little concentrated sulphuric acid by pouring it into an equal volume of water. *Water must never be poured into concentrated sulphuric acid.* Why?

(*c*) Place a splinter of wood in some concentrated sulphuric acid in a test tube.

(*d*) In a test tube, place about a cubic centimeter of sugar and add a few drops of concentrated sulphuric acid.

(*e*) Allow a crystal of copper sulphate to remain in some concentrated sulphuric acid in a test tube.

(*f*) Pass hydrogen sulphide through warm concentrated sulphuric acid. Explain the odor of sulphur dioxide. What is the precipitate?

(*g*) Heat a little sulphur in concentrated sulphuric acid in a test tube. Explain the formation of sulphur dioxide.

(*h*) Compare crystals of acid potassium sulphate with crystals of normal potassium sulphate. Make dilute solutions of each and test them with blue litmus paper.

Experiment 80. Fuming Sulphuric Acid. ¶ 81. (*a*) Examine some fuming sulphuric acid. Of what do the fumes consist?

(*b*) Heat a little solid ferric sulphate in a test tube. What is given off?

Experiment 81. Chlorides of Sulphuric Acid. ¶¶ **89, 90.** (*a*) Examine a little monochlor-sulphonic acid and a little sulphuryl chloride. Define the term " acid chloride."

(*b*) Add a few drops of water to a small quantity of monochlor-sulphonic acid in a test tube. Test sulphuryl chloride similarly.

Experiment 82. Persulphuric Acid. ¶ **91.** Describe the preparation of potassium persulphate.

(*a*) To one drop of a solution of manganese sulphate in a test tube, add 1 c.c. concentrated nitric acid, and 5 c.c. silver nitrate solution. Now add 3 g. solid potassium persulphate, and warm the mixture. How has the manganese ion been changed?

SODIUM THIOSULPHATE

¶ 93

Experiment 83. To a dilute solution of iodine in potassium iodide, add a few drops of starch solution, and then add drop by drop a solution of sodium thiosulphate until the blue color disappears. Explain.

Experiment 84. Iodometry. Clean a 50-c.c. burette by rinsing it with a mixture of concentrated sulphuric acid and potassium bichromate crystals. Obtain from the instructor a standard solution of sodium thiosulphate. After rinsing the burette, first with water and then with the thiosulphate solution, fill it with the sodium thiosulphate. Let the solution escape drop by drop until the curve of the meniscus is tangent to the zero mark. Obtain exactly 20 c.c. of a dilute iodine

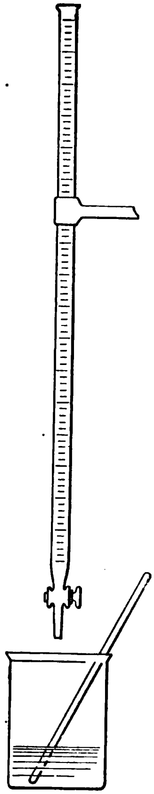


FIG. 32.

solution of unknown strength. Allow the thiosulphate solution to run from the burette into (titrate) the iodine solution, constantly stirring the mixture, until the original brown color changes to a light yellow. Now add starch solution and titrate until the blue color disappears. Take the reading of the burette.

Known thiosulphate standard. . . . . = ... *a* g. per c.c.
Number of c.c. thiosulphate used. . . . . = ... *b*
Number of g. thiosulphate used. . . . . = ... *ab*

Calculate the number of grams of iodine in the iodine solution.

$$Na_2S_2O_3 : I :: ab : x$$

$$158 : 127 :: ab : x$$

VOLUMETRIC ANALYSIS

Define standard solution, normal solution.

Examples. 1. Calculate the number of grams in 1 l. of the following normal solutions: HCl, NaOH, H_2SO_4, $Ca(OH)_2$, H_3PO_4, $AlCl_3$, H_2O_2, I.

2. 15.6 c.c. of a normal HCl solution were necessary to neutralize a certain NaOH solution 25 c.c. in volume. How many grams of NaOH did it contain? What was its strength in terms normal?

3. 22 c.c. of a normal NaOH solution were necessary to neutralize a certain H_2SO_4 solution 33 c.c. in volume. How many grams of H_2SO_4 did the solution contain? What was its strength in terms normal?

4. 20 c.c. of a .1 normal $KMnO_4$ solution * were decolorize

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* A normal $KMnO_4$ solution when used as an oxidizing agent contains 31.62 g. $KMnO_4$ to the liter.

by 35 c.c. of a certain H_2O_2 solution. (See ¶ 38.) How many grams of H_2O_2 did the solution contain? What was its strength in terms normal?

5. What was the strength in terms normal of the iodine solution analyzed in Exp. 84?

THERMOCHEMISTRY

¶¶ 97–100

Examples. 1. How many calories of heat are liberated when 20 g. of hydrogen are burned to liquid water?

2. What would the amount of heat be in 1, if the hydrogen had been burned to steam?

3. In the following reaction:

$$2KMnO_4Aq+5SnCl_2Aq+16HClAq= \\ 5SnCl_4Aq+2KClAq+MnCl_2Aq+8H_2O+3867K,$$

the heat of formation of $SnCl_2Aq=811.4K$, $HClAq=393.125K$, $SnCl_4Aq=1571.8K$, $KClAq=1011.5K$, $MnCl_2Aq=1250K$, $H_2O=68.35$. Calculate the heat of formation of $KMnO_4Aq$.

NITROGEN

¶¶ 105, 106

Experiment 85. (*a*) **Preparation from Air.** Cut a cavity in a flat cork so that a small crucible cover may be securely fastened to it. Float the cork in a basin of water and place a 4-mm. piece of yellow phosphorus on the crucible cover. Ignite the phosphorus and immediately hold a glass cylinder over it, so that the air in the cylinder is confined. See Fig. 33.

After the combustion has ceased, test the remaining gas with a burning splinter.

(*b*) **Preparation from Ammonium Nitrite.** Heat a concentrated solution of ammonium nitrite in a 100-c.c. flask fitted with an exit tube. Collect the gas over water. Test the gas with a burning splinter and with a well-ignited piece of phosphorus in a deflagrating spoon.

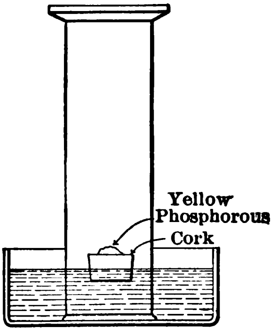

FIG. 33.

(*c*) **Preparation from Ammonium Bichromate.** Heat about 2 g. of ammonium bichromate in a 100-c.c. flask, collect the nitrogen over water, and test it as before.

THE ATMOSPHERE

¶¶ 107–109

Experiment 86.* Analysis of Air. Obtain from your instructor a gas burette; also a gas pipette containing phosphorus or an alkaline solution of pyrogallol. Draw exactly 100 c.c. of air into the burette and close the pinch cock.

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* If the class is large, this experiment may better be performed before the class by the instructor.

Connect the burette with the gas pipette and, after opening the pinch cock, if phosphorus is used, allow about 10 c.c. of air to enter the pipette. After a few moments, allow another 10 c.c. to enter, and so on, until all the air is in the pipette. (Too much air must not be allowed to enter the pipette at one time, since the phosphorus might take fire or melt.) Close the pinch cock, unfasten the pipette and shake it. After twenty minutes, connect the pipette with the burette and draw

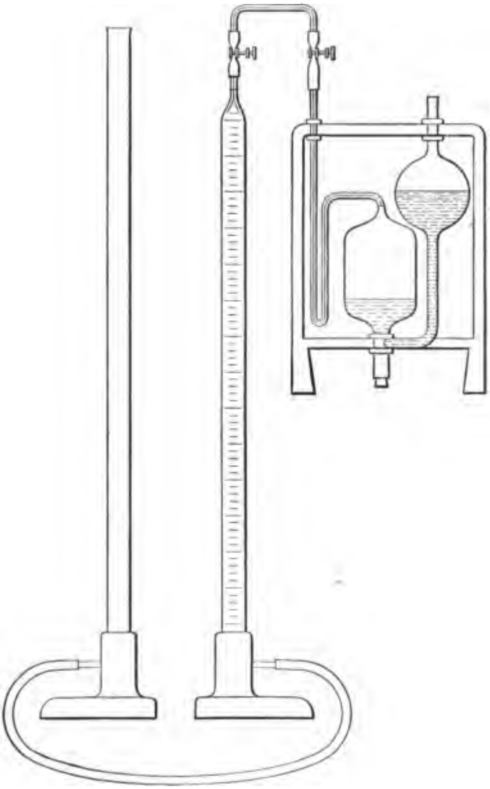

FIG. 34.

the air back, being careful that the capillary tube is full of water when the connection is made. After a few moments take the reading of the burette. What percentage of the air is oxygen?

AMMONIA

¶¶ 112, 113

Experiment 87. Preparation and Properties. (*a*) Powder about 10 g. each of ammonium chloride and of quick lime or soda lime. Mix the two substances well and heat them in a 200-c.c. flask fitted with a delivery tube. Collect several cylinders of the gas by air displacement, holding the cylinder upside down. Cover the cylinders with ground glass plates. Test the gas for solubility by opening a cylinder under water. Test the gas with moist red litmus paper. Plunge a glass rod moistened with concentrated hydrochloric acid into a cylinder of the gas.

(*b*) **From Distillation of Nitrogenous Organic Matter.** In a dry test tube, heat a small piece of gelatine under a hood. Test the escaping gas with moist red litmus paper. Do you detect the odor of ammonia?

Experiment 88. Liquid Ammonia. Examine a glass tube containing liquid ammonia. Describe the substance.

Experiment 89. Properties of Ammonia in Solution. Neutralize small quantities of dilute ammonia with hydrochloric acid, with nitric acid, and with sulphuric acid. Evaporate the solutions to a small volume on the water-bath or hot plate, and allow the dissolved substance to crystallize. What are the compounds thus formed?

HYDROXYLAMINE

¶ 118

Experiment 90. Properties of Hydroxylamine Hydrochloride. (*a*) Add 5 c.c. dilute sodium hydroxide to a dilute solution of copper sulphate, and then add 1 c.c. dilute hydroxylamine hydrochloride solution. Explain the formation of cuprous oxide.

(*b*) Add an excess of sodium hydroxide solution to a solution of ferrous sulphate in a test tube and warm the mixture. Add hydroxylamine hydrochloride until the ferrous hydroxide changes to brown. Dissolve the brown precipitate in hydrochloric acid, dilute the solution until it is light yellow, take 10 c.c. of this, add hydroxylamine hydrochloride, and allow the solution to stand for one hour. Explain what has occurred.

NITROUS OXIDE

¶ 119

Experiment 91. (*a*) **Preparation.** Connect a 100-c.c. retort containing about 5 g. of *dry* ammonium nitrate with a Woulff-bottle containing sodium hydroxide and a second Woulff-bottle containing ferrous sulphate solution. Heat the nitrate carefully and collect the nitrous oxide over warm water.

(*b*) **Properties.** Has the gas any odor? Any taste? Test the gas with a glowing splinter. Plunge a deflagrating spoon containing an ignited 3 mm. piece of phosphorus into a cylinder of the gas. Collect a mixture of about equal parts of nitrous oxide and hydrogen over warm water. Ignite the mixed gases.

NITRIC OXIDE

¶ 120

Experiment 92. (*a*) **Preparation.** Add 100 c.c. nitric acid (1 : 1) to a 500-c.c. flask containing about 20 g. of copper sheet or turnings, and collect the resulting gas over water under a hood. *Do not breathe the gas.*

(*b*) **Properties.** 1. Open a cylinder of nitric oxide under a hood.

2. Plunge a burning splinter into a cylinder of the gas.

3. Do the same with a well ignited small piece of phosphorus in a deflagrating spoon.

4. Do the same with burning sulphur.

5. Collect a cylinder of equal parts of hydrogen and nitric oxide and ignite the mixture.

6. To a cylinder of nitric oxide, add .5 c.c. of carbon bisulphide. Allow the bisulphide vapor and the gas to mix, and then ignite the mixture.

7. To a cylinder of nitric oxide, add about 50 c.c. of ferrous sulphate solution. Cover the cylinder with a glass plate and shake it.

Experiment 93. Other Methods for Making Nitric Oxide. (*a*) In a test tube, fitted with a delivery tube, boil equal parts of nitric acid, hydrochloric acid, and strong ferrous chloride solution. Collect the gas in a test tube over water.

(*b*) Similarly collect the gas formed when a little powdered potassium ferrocyanide, potassium nitrite, and acetic acid are heated together in a test tube.

NITROGEN DIOXIDE

¶ 122

Experiment 94. (*a*) **Preparation of Nitrogen Dioxide.** In a small retort heat about 25 g. of dry powdered lead nitrate and pass the gas through a test tube packed in a mixture of ice and salt. Collect by air displacement in cylinders any gas which may pass through the tube.

(*b*) **Properties of Nitrogen Dioxide.** 1. Add a little water to a test tube full of the gas, shake it, and see if the gas dissolves in water. Test the solution with blue litmus paper.

2. Allow the test tube containing the liquid nitrogen dioxide to grow warm gradually in the air. Notice any color changes that may take place.

3. Pour a little potassium iodide solution into a cylinder or test tube containing nitrogen dioxide.

OXYACIDS OF NITROGEN

¶¶ 124–127

Experiment 95. Nitrites. ¶ 126. (*a*) Heat about 10 g. of potassium nitrate in an iron crucible until the molten salt begins to give off oxygen. While the heating continues, sprinkle small quantities of granular lead into the molten mass. After a few minutes, allow the substance to cool, and dissolve it in water. Filter, and evaporate the solution on a water-bath to dryness.

(*b*) Test a little of the substance with sulphuric acid. How does it differ from potassium nitrate in this respect?

(*c*) To a few cubic centimeters of a concentrated solution of potassium nitrite, add a dilute solution of silver nitrate. What are the crystals which form?

(*d*) Calculate the number of grams of sulphuric acid equivalent to 2 g. of potassium nitrite. Dissolve 2 g. of nitrite in 200 c.c. of water. Dissolve the equivalent of sulphuric acid in 200 c.c. of water. Mix the two solutions. Test some of the mixture with dilute potassium permanganate solution. Explain.

Experiment 96. Nitric Acid. ¶ 127. (*a*) **Preparation.** Place about 10 g. of powdered potassium or sodium nitrate in a small retort and cover the salt with concentrated sulphuric acid. Place the retort on a sand bath, and support it by means of a ring stand so that the end of the neck extends into a 100-c.c. flask resting in a funnel held by a clamp. Allow cold water from the tap to run over the flask, through the funnel, and thence to the sink as in Fig. 21. Heat the retort gently and distil the nitric acid over into the flask.

(*b*) **Properties.** 1. Boil a little concentrated nitric acid in a test tube under a hood. Is the acid stable at the boiling temperature? If not, into what does it decompose?

2. Neutralize sodium hydroxide, potassium hydroxide, and calcium hydroxide solutions with nitric acid, evaporate the solutions to a small volume, and allow them to crystallize. What are the products?

3. Try the action of nitric acid, cold and hot, dilute and concentrated upon lead, copper, iron, zinc, and magnesium. Note carefully in each case how the acid acts.

4. To a piece of zinc in a test tube add 2 c.c. of dilute sulphuric acid and 2 drops of copper sulphate solution. As soon as hydrogen forms freely, add 1 c.c. of dilute nitric acid and note the effect. After a few moments, pour off the liquid, evaporate it to dryness, and heat the dry residue with a little concentrated sodium hydroxide solution. Do you recognize the odor of the gas that is given off ? Test the gas with moist

red litmus paper, being careful not to touch the paper to the sides of the tube. Explain the reaction.

5. Drop a small piece of glowing charcoal into a little hot fuming nitric acid in a test tube. Hold the tube away from the face, since the acid may spatter.

6. Immerse a piece of cloth in nitric acid for a few minutes. Wash the cloth in water, and examine it with respect to color, texture, etc.

Experiment 97. Nitrosyl-Sulphuric Acid. Pass dry sulphur dioxide into a Woulff bottle containing about 20 c.c. fuming nitric acid, the Woulff bottle being packed in ice. Spread the crystalline mass upon a porous plate by means of a glass rod. When the excess liquid has been absorbed, place the crystals in a dry glass stoppered bottle. In what experiment have you obtained similar crystals? In a test tube, add a little water to some of the crystals. Explain the formation of the red fumes.

Experiment 98. Nitrosyl Chloride. ¶ 129. Pass a mixture of dry chlorine and nitric oxide (dried by passing it through a calcium chloride tube) by means of a Y tube through a cylinder containing dry bone-black. Collect the gas by displacement of air under a hood. To a cylinder of gas, add a little water, and shake it. Test part of the liquid for hydrochloric acid by means of dilute silver nitrate

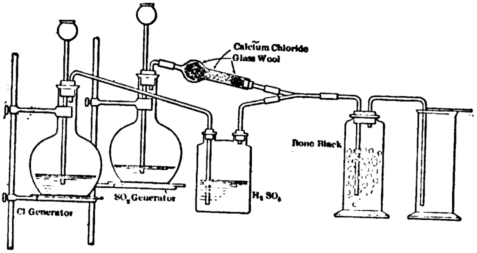

FIG. 35.

solution. To the remainder, add a few drops of dilute potassium permanganate solution. What causes the decolorization?

Experiment 99. Aqua Regia. In a test tube, warm gently three parts of concentrated hydrochloric acid and one part of concentrated nitric acid. Add a small piece of gold leaf to the liquid. Explain the solvent action.

PHOSPHORUS

¶¶ 131–135

Experiment 100. Occurrence. Examine and describe specimens of phosphorite, wavellite, apatite, and bone-ash.

Experiment 101. Physical Properties of Phosphorus. (*a*) Examine and describe specimens of yellow and red phosphorus. *Use great care in handling the yellow variety, since a burn from it heals very slowly. Always handle it with forceps.*

(*b*) Heat a 3 mm. piece of yellow phosphorus under water in a test tube. Does it melt? Try red phosphorus in the same way.

(*c*) To a *dry* test tube containing about 1 c.c. carbon bisulphide, add a 4 mm. piece of dry yellow phosphorus. When the phosphorus has dissolved, pour the solution upon a piece of filter paper under a hood. Try the solubility of red phosphorus in carbon bisulphide.

(*d*) Into a glass tube of about 5 mm. bore and 6 cm. long, and closed at one end, introduce a 4 mm. piece of dry phosphorus. Add a small crystal of iodine. Seal the open end of the tube in the blast lamp flame. Place the sealed tube in an air bath, suspend a thermometer in it, and heat the whole to 200° for a few minutes. When the tube is cool, open it, remove the contents, rinse them upon a piece of filter paper with a little carbon bisulphide, and compare the product with a specimen of red phosphorus.

Experiment 102. Chemical Properties of Phosphorus. The ease with which phosphorus may be oxidized has been observed.

(*a*) To a test tube arranged as in Exp. 47 (*b*), and containing a little bromine, add a 3 mm. piece of yellow phosphorus. What is formed?

(*b*) Similarly try the effect of fuming nitric acid upon phosphorus. Preserve the solution, and, after Exp. 109 has been performed, test it according to Exp. 109 (*b*), 6.

(*c*) Place a clean piece of yellow phosphorus in a test tube containing copper sulphate solution. Explain.

(*d*) To what is the odor of yellow phosphorus due? See Exp. 42 (*a*).

HYDROGEN PHOSPHIDE

¶ 136

Experiment 103. Preparation of Hydrogen Phosphide. (*a*) Heat a little solid phosphorous acid in a test tube under a hood. Explain the formation of phosphine.

(*b*) In a 100-c.c. flask place a 5 mm. piece of phosphorus in a solution of concentrated potassium hydroxide to which 30 c.c. of alcohol have been added. Fit the flask with a rubber stopper carrying a delivery tube. Heat the mixture and collect the gas over water, performing the experiment under a hood. What is the reason for adding alcohol to the potassium hydroxide?

(*d*) To a few small pieces of calcium phosphide in a test tube, add a little hydrochloric acid under the hood. Explain the formation of hydrogen phosphide.

Experiment 104. Properties of Phosphine. State the physical properties of the gas. Plunge a hot glass rod into a cylinder of the gas. Add a solution of copper sulphate to a cylinder of the gas. Compare hydrogen phosphide with ammonia with respect to its basic properties.

PHOSPHORUS TRICHLORIDE

¶ 140

Experiment 105. Examine a little phosphorus trichloride. Why does it fume in the air? Add a little water to a small quantity of phosphorus trichloride in a test tube under the hood. What is formed?

PHOSPHORUS PENTACHLORIDE

¶ 141

Experiment 106. Examine and describe a specimen of phosphorus pentachloride. To a small quantity of phosphorus pentachloride in a test tube under a hood, add a small quantity of water. Compare this product with the substance to be studied in the next experiment. In another test tube try the effect of a large quantity of water. In a large long test tube heat about 3 g. phosphorus pentachloride over the Bunsen flame under the hood. Can you distinguish any yellow color on looking down the tube? If so, explain its presence. If you wish to purify crude phosphorus pentachloride, how would you do so?

PHOSPHORUS OXYCHLORIDE

¶ 142

Experiment 107. Examine and describe a specimen of phosphorus oxychloride. Try the effect of water upon the substance under the hood.

PHOSPHORUS PENTOXIDE

¶ 144

Experiment 108. Whan phosphorus burns in air or oxygen what compounds are formed? Examine and describe a specimen of phosphorus pentoxide. Try the effect of adding a little water to a small quantity of phosphorus pentoxide on a watch-glass. Test the solution with blue litmus paper.

ACIDS OF PHOSPHORUS

¶ 145

Experiment 109. Phosphoric Acid. (*a*) **Preparation.** Weigh out 2 g. calcium phosphate. Calculate the amount of concentrated sulphuric acid required to liberate the phosphoric acid. Weigh out this amount of sulphuric acid, dilute it with twenty times its volume of water, add the diluted acid to the phosphate, and stir the mass thoroughly. Filter off the calcium sulphate, and evaporate the filtrate to dryness on the water-bath.

(*b*) **Properties of the Acid and of its Salts.** 1. Examine the properties of the acid as to taste (dilute solution) and action upon blue litmus paper.

2. Try the solubility of calcium phosphate in the dilute mineral acids. Explain.

3. To a solution of acid sodium phosphate, add an excess of a dilute solution of silver nitrate. Filter off the precipitate (characteristic of phosphates) and test the filtrate with blue litmus paper. Explain. Now add a pinch of powdered sodium acetate to the filtrate. Explain the formation of more yellow precipitate.

4. Dissolve small quantities of primary, secondary and tertiary potassium phosphates in small amounts of water, and test each solution with litmus paper, blue and red. Explain the reactions.

5. Make a solution of acid sodium phosphate alkaline with ammonia, then add dilute ammonium chloride and magnesium chloride. What is the precipitate and what use is made of it in analytical chemistry?

6. To a nitric acid solution of calcium phosphate, add an excess of a nitric acid solution of ammonium molybdate and warm the mixture to about 60° C. What is the yellow precipitate which forms, and what is its value in analytical chemistry?

PYROPHOSPHORIC ACID

¶ 147

Experiment 110. Heat a few crystals of dehydrated acid sodium phosphate in a hard glass tube until the mass is molten and no more water is given off. Dissolve the salt in water, and to a portion add dilute silver nitrate. Compare the precipitate with the one obtained with silver nitrate and acid sodium phosphate.

METAPHOSPHORIC ACID

¶ 148

Experiment 111. Heat some crystals of ammonium acid phosphate in a hard glass tube until no more ammonia and water are given off. Dissolve the product in cold water. Test the solution with blue litmus paper. To a portion add a solution of egg albumen. To another portion, add dilute barium chloride. How can you distinguish metaphosphoric acid from pyrophosphoric acid?

PHOSPHOROUS ACID

¶ 151

Experiment 112. To about 5 c.c. of phosphorus trichloride in a small beaker, add under a hood about 10 c.c. water. What gas is given off? Evaporate the liquid to dryness. What is the solid obtained? Heat a portion of it in a hard glass tube and explain the decomposition. Dissolve the rest in water. To a portion add dilute silver nitrate solution, or dilute mercuric chloride solution. To another portion add a solution of sulphurous acid. What is the most characteristic property of phosphorous acid? How many replaceable hydrogen atoms has the phosphorous acid molecule?

HYPOPHOSPHOROUS ACID

¶ 152

Experiment 113. To a solution of potassium hypophosphite, add dilute mercuric chloride, to a second add sulphurous acid, to a third add dilute copper sulphate. How can you distinguish between phosphites and hypophosphites? How many replaceable hydrogen atoms has the hypophosphorous acid molecule?

PHOSPHORUS PENTASULPHIDE

¶ 153

Experiment 114. Examine and describe a specimen of phosphorus pentasulphide. Warm a little of the substance with water under a hood. What gas is given off? Test the solution, filtered, if necessary, for phosphoric acid.

ARSENIC

¶ 155

Experiment 115. Occurrence of Arsenic. Examine and describe specimens of native arsenic, orpiment, realgar, arsenopyrite, and cobaltite.

Experiment 116. Preparation. (*a*) Heat a little arsenopyrite in a hard glass tube.

(*b*) Heat a little arsenic oxide mixed with charcoal in a hard glass tube. What is the black mirror?

Experiment 117. Properties of Arsenic. Place a small piece of arsenic in a hard glass tube and heat it. Describe the sublimed substance. In what allotropic forms is arsenic known? Heat a small piece of arsenic by means of the blow-pipe flame under a hood. What is the white smoke?

HYDROGEN ARSENIDE

¶¶ 156, 157

Experiment 118. Marsh Test. Set up a hydrogen generator (not a Kipp generator) under a hood, and pass a slow stream of the gas through a tube containing calcium chloride and connected with a long glass tube drawn out to 1 mm. opening. When all air has been expelled from the apparatus,* ignite the hydrogen, and hold a cold porcelain crucible cover in the flame. Now introduce a few drops of any solution containing arsenic, and again introduce the crucible cover into the flame. Does the flame change color? What is deposited on the cover? Try the solubility of the deposit in sodium hypochlorite solution. Heat the glass tube with the flame at about the middle. Note the sublimate which forms in the cool part of the tube.

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* Caution. See Exp. 18 (*e*).

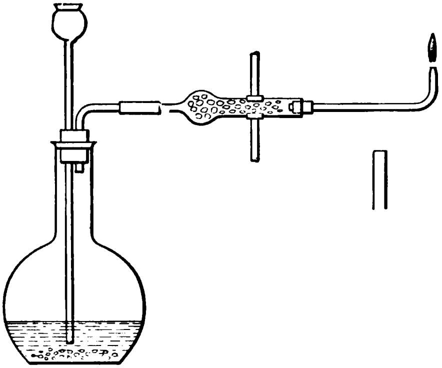

FIG. 36.

Experiment 119. Gutzeit's Test. Hold a piece of filter paper moistened with a 50 per cent solution of silver nitrate over a test tube containing zinc and dilute sulphuric acid, together with a few drops of an arsenic solution. Place a plug of cotton in the upper part of the tube to protect the paper from being spattered by the effervescing solution.

ARSENIOUS OXIDE

¶ 159

Experiment 120. Properties. Examine and describe a specimen of arsenious oxide. Place a small quantity in a tube closed at one end and heat it in the flame. Examine the sublimate with a magnifying glass. Do any other allotropic forms of arsenious oxide exist? Warm a small quantity of arsenious oxide with water, filter, and test the filtrate with blue litmus paper. Test its solubility in hot hydrochloric acid under the hood. Test the solubility of the oxide in sodium hydroxide, and in sodium carbonate solution.

ARSENIC OXIDE

¶ 160

Experiment 121. Properties. Examine and describe a specimen of arsenic oxide. Heat a small quantity in a hard glass tube and test for oxygen. Warm a small quantity with water and test the solution with blue litmus paper.

ARSENIOUS ACID

¶ 161

Experiment 122. (*a*) Add a dilute solution of silver nitrate to a dilute solution of sodium arsenite. Add dilute ammonia to the precipitate.

(*b*) Dissolve a little arsenious oxide in sodium hydroxide solution. Then make the solution acid with hydrochloric acid and finally alkaline with acid sodium carbonate. Add this solution, drop by drop, to a dilute iodine solution. Explain the decolorization of the iodine solution.

ARSENIC ACID

¶ 162

Experiment 123. (*a*) To a solution of potassium arsenate in a 100-c.c. flask, add magnesia mixture. Shake the flask, and examine the precipitate with a magnifying glass. What is the chemical constitution of the precipitate? Compare the precipitate with the one obtained in the same way, using a phosphate instead of an arsenate.

(*b*) Add dilute silver nitrate solution to a solution of potassium arsenate. Test the solubility of the precipitate in ammonia. Compare the precipitate with that obtained in the same way, using a phosphate instead of an arsenate.

(*c*) To a solution of potassium arsenate, strongly acid with concentrated nitric acid, add a nitric acid solution of ammonium molybdate. What is the precipitate? Compare the precipitate with that obtained in the same way, using a phosphate instead of an arsenate.

SULPHIDES OF ARSENIC

¶¶ 163, 164

Experiment 124. (*a*) **Arsenious Sulphide.** Into a solution of potassium arsenite, pass a stream of hydrogen sulphide for about two minutes. Does the appearance of the solution change? Now add a little concentrated hydrochloric acid. What is the yellow precipitate? Filter off the precipitate and try its solubility in not too much ammonium sulphide. To the solution thus obtained, add a little concentrated hydrochloric acid. What is the precipitate?

(*b*) **Arsenic Sulphide.** Into an acid solution of potassium arsenate, pass a stream of hydrogen sulphide for about ten minutes. Treat the precipitate as in (*a*), and explain the various reactions.

ANTIMONY

¶ 165

Experiment 125. (*a*) **Occurrence.** Examine and describe a specimen of stibnite.

(*b*) **Stibine.** ¶ **166.** Make stibine just as you made arsine in Exp. 118. How does stibine differ from arsine?

2. Test for stibine just as you tested for arsine by means of Gutzeit's test.

HALOGEN COMPOUNDS OF ANTIMONY

¶ 167

Experiment 126. (*a*) **Antimony Trichloride.** Powder about 5 g. of stibnite, and treat the powder under a hood with concentrated hydrochloric acid, warming the mixture until all the stibnite is dissolved and all hydrogen sulphide is driven off. Dilute the solution as long as no permanent precipitate appears, filter, and evaporate it to dryness on the water-bath. What is the crystalline mass? Place a little in a test tube and warm it gently. Add a little strong hydrochloric acid to a little of the substance in a test tube and warm it until the crystals dissolve. Now add water, drop by drop, until a white precipitate forms. What is the precipitate?

(*b*) **Antimony Pentachloride.** Pass chlorine over molten antimony trichloride in an apparatus like that used in Exp. 75, for making sulphur monochloride. Place some of the liquid thus obtained in a test tube and, after adding a few drops of water, allow the test tube to stand corked until the next day. Boil a little of the liquid with water in a test tube.

ANTIMONY TRIOXIDE

¶ 168

Experiment 127. Antimony Trioxide. (*a*) Examine and describe a specimen of antimony trioxide.

(*b*) Try the solubility of antimony trioxide in the mineral acids, in tartaric acid, and in the alkalis.

Experiment 128. Meta-antimonious Acid. To a solution of antimony trichloride, add a solution of sodium carbonate. Filter off the white precipitate, and try its solubility in acids and in alkalis. What is the graphic formula for meta-antimonious acid? for antimonyl chloride? for antimonyl sulphate? for antimonyl potassium tartarate?

ANTIMONIC ACID AND ANTIMONY PENTOXIDE

¶ 168

Experiment 129. Antimonic Acid. Powder about 5 g. of antimony and treat it with about 20 c.c. of hot concentrated nitric acid. When all the antimony has been oxidized, add about 100 c.c. of water and filter off the white substance. Wash the precipitate free from nitric acid on the filter. Try the solubility of the antimonic acid in sodium hydroxide solution and in hydrochloric acid.

Experiment 130. Antimony Pentoxide. Heat a little antimony pentoxide in a hard glass tube, testing for the evolution of oxygen. What is the color while hot of the remaining solid? What is it?

SULPHUR COMPOUNDS OF ANTIMONY

¶ 169

Experiment 131. Sulphides of Antimony. **¶ 169.** (*a*) Pass hydrogen sulphide gas into a hydrochloric acid solution of antimony trichloride. Filter off the precipitate, and to a portion add enough of a solution of yellow ammonium sulphide to dissolve it. To the solution thus obtained add concentrated hydrochloric acid. Explain the reaction. To a second portion of the original precipitate, add hydrochloric acid. Dry a third portion, and melt the dried product in a hard glass tube. Compare the melted sulphide with stibnite.

(*b*) Pass hydrogen sulphide into a solution of potassium antimoniate, slightly acid with hydrochloric acid. Filter off the precipitate and treat portions with ammonium sulphide and with hydrochloric acid as in (*a*).

(*c*) Boil 10 g. of antimony trisulphide with a solution of 10 g. of sodium hydroxide and 3 g. of sulphur dissolved in 20 c.c. of water. Filter the solution and allow it to crystallize. What is the chemical name for the crystals? What is the commercial name? Let a crystal remain in the open air for several days, and note any change which may occur.

BISMUTH

¶¶ 170–174

Experiment 132. (*a*) **Occurrence, Preparation, and Properties of Bismuth.** Examine and describe a specimen of bismuth glance. Heat a piece upon charcoal, using the blowpipe flame. Examine the metallic bead thus obtained, and compare it with a specimen of metallic bismuth. State the physical properties of the metal. What is the brown incrustation formed on the charcoal? Try the action of dilute and concentration mineral acids upon bismuth.

(*b*) **Bismuth Chloride. ¶ 171.** Dissolve a little powdered bismuth in aqua regia, and evaporate the solution to dryness. Dissolve the crystalline solid in dilute hydrochloric acid, and add water drop by drop to the acid solution. Explain the formation of the white precipitate.

(*c*) **Bismuth Trioxide. ¶ 172.** Examine and describe a specimen of bismuth trioxide. Heat a little solid bismuth nitrate in glass tube, and compare the product with bismuth trioxide. To a boiling solution of bismuth nitrate, add boiling potassium hydroxide solution. Examine the precipitate with a magnifying glass. Try the solubility* of bismuth trioxide in alkalis and in acids. Is it a basic or an acid oxide?

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* To try the solubility of any precipitate, filter it off, wash it, place a little of it in a clean test tube, and then add the solvent.

(*d*) **Bismuth Hydroxide.** **¶ 173.** To a solution of bismuth nitrate in a test tube, add dilute sodium hydroxide. Try the solubility of the white precipitate in excess of sodium hydroxide and in ammonia. Try the solubility in mineral acids. Is bismuth hydroxide acid or basic?

(*e*) **Bismuth Nitrate and Subnitrate.** **¶ 173.** Examine a crystal of bismuth nitrate. Add about twenty parts of water to the crystal, and boil the mixture. What is the precipitate?

(*f*) **Bismuth Trisulphide.** **¶ 174.** Pass hydrogen sulphide into an acid solution of bismuth nitrate. Filter off the precipitate and try its solubility in acids and in alkaline sulphides.

CARBON

¶¶ 176–179

Experiment 133. Physical and Chemical Properties. Examine the different forms of carbon that may be found in the laboratory, and make a table showing their physical and chemical properties so far as you can learn them through experiment or reading. See Exp. 14, (*b*).

METHANE

¶ 180

Experiment 134. (*a*) Powder 2 g. of fused sodium acetate and 2 g. of sodium hydroxide. Mix these with 8 g. of powdered quick lime (or 10 g. of powdered soda lime may be used with 2 g. of sodium acetate). Heat the mixture in a 100-c.c. flask fitted with exit and delivery tubes. Collect the gas over water. Ignite a cylinder of the gas. Ignite a cylinder containing 1 part of the gas and 9 parts of air. Is the gas heavier or lighter than air?

(*b*) Allow water to drop by means of a dropping funnel upon a small quantity of aluminum carbide in a flask fitted with an exit tube, and collect the resulting methane over water.

Experiment 135. Acetylene. ¶ 181. Place a few pieces of calcium carbide in a 100-c.c. flask, and fit the flask with a dropping funnel and a delivery tube. Allow water to drop on to the calcium carbide in the flask, and collect the acetylene over water. Test the gas as to odor, solubility, combustibility, and explosibility when mixed with air.

CARBON TETRACHLORIDE

Experiment 136. Place ½ c.c. of carbon tetrachloride in each of several test tubes and test it as to solubility in water, as to volatility and as to inflammability. Has the substance any odor? Test the solubility of vaseline in carbon tetrachloride. Would carbon tetrachloride be useful in removing grease stains?

CARBON MONOXIDE

¶ 182

Experiment 137. (*a*) **Preparation from Carbon Dioxide.** Fill a 50-cm. long piece of gas pipe with pieces of charcoal, and fit the pipe with corks carrying entrance and exit tubes. Heat the pipe to redness by means of two blast lamps, and pass a slow stream of carbon dioxide over the red hot charcoal. Collect the gas over water. Shake a cylinder of the gas with sodium hydroxide solution to remove any unchanged carbon dioxide, and ignite the remaining gas.

(*b*) **Preparation of Water Gas.** Make water gas by passing steam over red hot charcoal in a gas pipe as in (*a*).

(*c*) **Preparation from Oxalic Acid.** Place about 10 g. of oxalic acid in a 200-c.c. flask and cover the crystals with concentrated sulphuric acid. Fit the flask with a cork carrying a safety tube and an exit tube. Heat the flask and pass the evolved gas, first through an empty Woulff-bottle and then through a second containing sodium hydroxide solution. Collect the gas over water. Perform the experiment under the hood.

Experiment 138. Properties of Carbon Monoxide. (*a*) Is the gas soluble in water? Does it burn? Pour about 30 c.c. ammoniacal cuprous chloride solution into a cylinder of the gas and shake it. Is carbon monoxide soluble in the solution?

(*b*) Pass a stream of carbon monoxide from the apparatus of Exp. 137 (*c*), over hot copper oxide in a hard glass tube under a hood. Is carbon monoxide a reducing agent?

CARBON DIOXIDE, CARBONIC ACID, AND CARBONATES

¶ 184

Experiment 139. (*a*) **Preparation from Carbon. See Exp. 14** (*b*).

(*b*) **Preparation from Acid Sodium Carbonate.** Heat about 2 g. of acid sodium carbonate in a test tube and collect the evolved gas by displacement of air in another test tube. Add 2 c.c. of clear lime water to the test tube and shake it.

(*c*) **Preparation from Magnesite.** Perform a similar experiment using magnesite in a hard glass tube.

(*d*) **Preparation from Marble and Hydrochloric Acid.** Treat a few pieces of marble in a test tube with dilute hydrochloric acid, collecting the gas and testing it as before.

(*e*) Collect several cylinders of the gas from a Kipp apparatus containing marble and hydrochloric acid.

Experiment 140. Properties. (*a*) Make a siphon of glass tubing about 1 sq. cm. in cross-section and siphon the gas from one cylinder to another. Test for the presence of carbon dioxide in the second cylinder.

(*b*) Collect some solid carbon dioxide by opening carefully the stop-cock of a slightly inverted cylinder of liquid carbon dioxide so that the solid which forms collects in a cloth bag which has been tied securely over the valve. Explain the formation of the solid carbon dioxide. Place a little mercury in a porcelain dish, cover the mercury with solid carbon dioxide, and then add a little ether. Have no open flame near. Why does the mercury freeze?

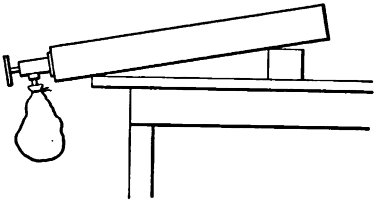

FIG. 37.

(*c*) Lower an ignited candle into a cylinder of carbon dioxide.

(*d*) Lower a well ignited piece of magnesium ribbon into a cylinder of carbon dioxide.

(*e*) Shake a cylinder of carbon dioxide with water. Taste the solution. Test the solution with blue litmus paper.

(*f*) Try the effect of various acids upon small quantities of sodium carbonate in test tubes.

(*g*) Test a solution of sodium carbonate with red litmus paper. Explain.

(*h*) Add a little sodium carbonate to dilute solutions of copper sulphate, mercuric chloride, bismuth nitrate, and lead nitrate.

(*i*) Pass carbon dioxide into lime water until the precipitate which first forms dissolves. Heat the solution until the precipitate reappears. Explain.

FLAME

¶ 186

Experiment 141. (*a*) **Reverse Flame.** Fit a lamp chimney (globe form), or an Argand chimney covered with a perforated square of asbestos as in Fig. 38, with a two-holed cork at its lower end. Fit the cork with a 5 mm. glass tube bent at right angles and a straight 10 mm. tube extending about half-way up the chimney. Remove the chimney, allow gas to pass through the small glass tube so that when ignited it gives a small flame. Replace the chimney, and increase the supply of gas gradually. At a certain moment the flame will be extinguished and a large flame will appear at the end of the larger tube. Explain.

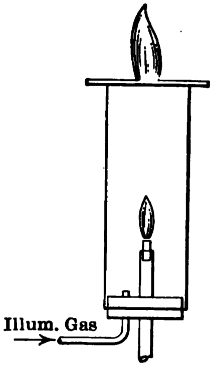

FIG. 38.

(*b*) **Combustion of Potassium Chlorate in Illuminating Gas.** The lamp chimney is fitted at its lower end with a one-holed cork with an entrance tube through which illuminating gas is entering. The chimney is covered with a square of asbestos having a 2 cm. opening, and the gas is ignited. Lower into the chimney a deflagrating spoon containing a little potassium chlorate heated to the point where it gives off oxygen. Explain.

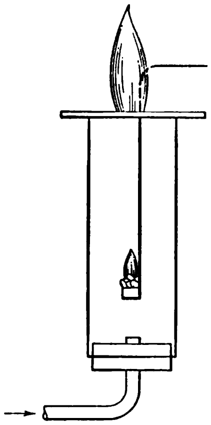

FIG. 39.

(*c*) **Luminosity of Flame.** Arrange the Bunsen burner so that it gives a non-luminous flame. Rub two sticks of carbon together and then strike them together near the flame. Explain the luminosity of the ordinary gas flame.

(*d*) **Principle of the Miners' Safety Lamp.** Hold a piece of wire gauze above the top of an open but unlighted Bunsen burner. Light the gas above the gauze. Why does the gas burn only above the gauze? Remove the gauze sideways, so that the flame burns as usual. Now bring the gauze gradually down upon the flame. Why does the gas burn only below the gauze?

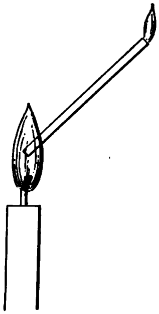

FIG. 40.

(*e*) **Zones of a Flame.** 1. Ignite a large candle. Insert one end of a 2-mm. tube into the center of the candle flame just above the wick as in Fig. 40. Ignite the gas escaping from the other end of the tube.

2. Press a piece of cardboard down upon the flame of a Bunsen burner for a few moments. Examine the paper and, from the appearance of the cardboard, draw conclusions concerning the interior of the flame.

Hold a platinum wire across the flame and examine its appearance carefully.

SILICON

¶ 190

Experiment 142. Occurrence. Examine and describe specimens of quartz, tridymite, onyx, opal, jasper, sandstone, feldspar, etc.

Experiment 143. Preparation of Silicon. (*a*) Mix 20 g. powdered quartz with 10 g. magnesium powder in an iron crucible and heat the mixture with a blast lamp until the action commences. After the reaction mixture has cooled, remove it from the crucible, pulverize and treat it with dilute hydrochloric acid. Describe the silicon residue.

(*b*) Mix 4 g. aluminum filings, 5 g. of sulphur, and 3.6 g. of powdered quartz, and place the mixture on a square of asbestos under the hood. Ignite the mass, and when the reduced silicon is cold, purify it by means of hydrochloric acid as in (*a*).

Experiment 144. Properties of Silicon. Try to oxidize silicon with the flame of the blowpipe. Make a cavity in a piece of paraffine and place a little silicon and hydrofluoric acid in the cavity (under the hood). Heat a little silicon with concentrated potassium hydroxide in a test tube fitted with a cork and a delivery tube. Is any hydrogen given off?

HYDROGEN SILICIDE

¶ 191

Experiment 145. Preparation of Hydrogen Silicide. Mix 6 g. magnesium powder and 10 g. powdered quartz. Heat the mixture in an iron crucible and keep the crucible covered with a piece of asbestos paper. When the magnesium silicide thus formed is cool, purify it by washing it in water eight or ten times. Treat a little of the magnesium silicide with hydrochloric acid in a test tube. What is the gas which is formed? Why does the gas take fire in the air?

SILICON TETRAFLUORIDE

¶ 192

Experiment 146. Action of Hydrofluoric Acid upon Silica.*

(*a*) Treat a little pure silica in a platinum crucible with hydrofluoric acid and a drop of concentrated sulphuric acid.

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* If the class is large, it will probably be necessary to omit this experiment on account of a lack of platinum.

Heat the crucible on the water-bath under the hood. When only sulphuric acid remains, drive off the acid by means of the Bunsen flame. Does anything remain in the crucible? How would you treat a silicate insoluble in acid, in order to bring it into solution?

(*b*) **Action of Water upon Silicon Tetrafluoride.** Mix equal parts of sand and powdered fluorspar in a 100-c.c. flask with enough concentrated sulphuric acid to make a thin paste. Fit the bottle with a cork, carrying a wide delivery tube bent twice at right angles. Let the opening of the delivery tube dip under a layer of mercury in the bottom of a test tube full of water. Heat the flask in a beaker of water. Note the action of the gas bubbles upon the water as they ascend to the surface. What is the white film which is formed around them? Test the water with litmus paper. What causes the acidity?

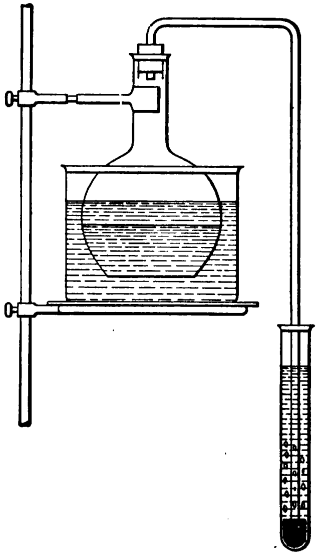

FIG. 41.

SILICON DIOXIDE AND SILICIC ACID

¶¶ 193, 194

Experiment 147. (*a*) To a 10-c.c. solution of water glass in a beaker, add enough hydrochloric acid to make the solution acid. Place half of the solution in another beaker, and allow it to stand. Evaporate the remainder to dryness, heat the residue for twenty minutes at a temperature of about 130°, then add 50 c.c. of water, filter, wash, and ignite the moist filter in a porcelain crucible. Mix a pinch of the pure silica thus obtained with an excess of dry potassium carbonate and fuse the mixture on a platinum foil. Is silica soluble in fused alkalis?

(*b*) Pour a little water glass into an excess of concentrated hydrochloric acid. How could the soluble silicic acid be separated from the solution?

COLLOIDS

¶ 196

Experiment 148. (*a*) **Hydrosol. Hydrogel.** To about 1 g. gelatine in a test tube, add 10 c.c. water and boil until the gelatine dissolves. Now cool the test tube under the water tap. Define hydrosol, hydrogel.

(*b*) **Diffusibility through a Hydrogel.** Make a hydrogel of agar-agar, and let it stand in a test tube containing an ammoniacal copper sulphate solution. Notice whether or not the blue color diffuses into the hydrogel.

(*c*) **Nondiffusibility of Colloids.** Try a similar experiment with the agar-agar hydrogel, using colloidal Prussian Blue made by adding a solution of ferric chloride to a solution of potassium ferrocyanide.

(*d*) **Colloidal Arsenious Sulphide.** Reread Exp. 124 (*a*).

TIN

¶ 198

Experiment 150. (*a*) **Occurrence.** Examine and describe a specimen of cassiterite.

(*b*) **Extraction.** Heat a little cassiterite or tin oxide upon charcoal by means of the reducing flame of the blowpipe. Examine and describe the metallic bead thus obtained.

(*c*) **Action of Acids and Alkalis upon Tin.** Try the action of the various mineral acids, concentrated and dilute, upon tin. Try the effect of hot, strong, sodium hydroxide solution upon tin.

STANNOUS COMPOUNDS

¶ 200

Experiment 151. (*a*) **Stannous Chloride. ¶ 200. Action of Water and Air on Stannous Chloride.** Dissolve a few grams of stannous chloride in a few cubic centimeters of water containing a drop of hydrochloric acid. Filter the solution until it is clear, and allow it to stand for some days in contact with the air. Explain the formation of a white precipitate.

(*b*) **Stannous Oxychloride.** Add water to a few crystals of stannous chloride in a test tube. Why can you not obtain a clear solution?

(*c*) **Reducing Action of Stannous Chloride.** 1. Dissolve 2 g. of stannous chloride in 10 c.c. hydrochloric acid (1 : 1). To a little mercuric chloride solution in a test tube, add a few drops of the stannous chloride solution, and boil the mixture. Explain the formation of the white precipitate which becomes gray on boiling.

2. Test solutions of potassium permanganate, potassium bichromate, and ferric chloride, with acid solutions of stannous chloride.

(*d*) **Stannous Hydroxide.** To a solution of stannous chloride, add dilute sodium carbonate. To part of the precipitate, add hydrochloric acid, and to another, add dilute sodium hydroxide. What can you say concerning the ionization of stannous hydroxide?

(*e*) **Stannous Oxide.** Place a small quantity of stannous hydroxide, prepared as in (*d*) in a hard glass tube containing an entrance and exit tube. Displace the air in the tube by means of a stream of dry carbon dioxide. Then heat the tube until all water is driven off. What is formed?

(*f*) **Stannous Sulphide.** Precipitate a solution of stannous chloride by means of a stream of hydrogen sulphide. Add ammonium sulphide to a part of the precipitate. Does it dissolve? Add yellow ammonium sulphide to another portion of the precipitate. Does it dissolve? If so, add hydrochloric acid to the solution. Explain the reactions.

STANNIC COMPOUNDS

¶ 201

Experiment 152. (*a*) **Stannic Chloride.** 1. Place a few grams of tin in a 100-c.c. distilling flask under a hood. Pass dry chlorine over the tin by means of an entrance tube extending almost to the bottom of the flask. Heat the tin so as to keep it melted and collect the stannic chloride in a receiver, fitted to the exit tube of the distilling flask.

2. Add a few drops of water to 1 c.c. of stannic chloride in a test tube. Cork the tube and let it stand.

3. Boil a little stannic chloride with water in a test tube and compare the reaction with that obtained in (*d*).

(*b*) **Stannic Oxide.** Make stannic oxide by heating a little tin in air in a crucible.

(*c*) **Stannic Acid.** Add dilute ammonia to an aqueous solution of stannic chloride. Filter off the precipitate and wash it thoroughly. Test the moist precipitate with blue litmus paper. Try the solubility of the stannic acid, thus obtained, in acids and in alkalis.

(*d*) **Metastannic Acid.** To a beaker containing about 1 g. of mossy tin, add about 5 c.c. concentrated nitric acid under a hood. When the action is ended, dilute the white precipitate with water, filter and wash it thoroughly. Test the solubility of the metastannic acid in sodium hydroxide. Boil a little in hydrochloric acid and test the solution with stannous chloride. What is the probable formula for metastannic acid? See ¶ **201.**

(*e*) **Stannic Sulphide. ¶ 202.** Pass hydrogen sulphide through an acid solution of stannic chloride. Test the solubility of the stannic sulphide in ammonium sulphide. If it dissolves, add hydrochloric acid to the solution. Explain.

LEAD

¶¶ 203–206

Experiment 153. (*a*) **Occurrence.** Examine and describe specimens of galena, cerussite, crocoite, and any other lead minerals that may be at hand.

(*b*) **Extraction.** Heat a little galena upon charcoal by means of a reducing blowpipe flame. Examine and describe the metallic bead thus obtained.

(*c*) **Properties.** 1. Try the action of the various mineral acids, dilute and concentrated, upon granular lead. Try the action of acetic acid.

2. In a solution of lead nitrate, suspend a strip of zinc. Explain the precipitation of lead by zinc.

3. Heat a few grams of lead tartrate in a test tube until no more organic fumes are given off. Close the tube with a rubber stopper and allow it to cool. When the tube is almost cool, open it, and pour out the contents. Explain.

Experiment 154. Oxides of Lead. ¶ 204. (*a*) **Lead Monoxide.** 1. Heat a few crystals of lead nitrate in a test tube until no fumes are given off.

2. Saturate a hot potassium hydroxide solution with lead oxide, and set the solution aside to crystallize. Preserve for (*c*) 5, and (*d*).

(*b*) **Lead Hydroxide.** Add sodium hydroxide to a dilute lead nitrate solution. Filter off the precipitate, and wash it thoroughly. Test the precipitate with moist red litmus paper. Try the solubility in sodium hydroxide solution and in ammonia.

(*c*) **Lead Peroxide.** 1. To a solution of lead nitrate, add a solution of sodium hypochlorite and boil the mixture. Filter the precipitat , dry it, and compare the product with lead peroxide.

2. Heat a little lead peroxide in a glass tube and test for the evolution of oxygen. Note any color changes.

3. Heat a little lead peroxide with concentrated sulphuric acid in a test tube, and notice the evolution of oxygen. When the acid is cold, dilute it. What is the precipitate?

4. Heat a little lead peroxide with concentrated hydrochloric acid and test for chlorine.

5. Saturate a little hot concentrated caustic potash solution with lead peroxide. Dilute the solution, and add a similar solution of lead oxide in caustic potash.

In the light of the above facts, discuss the chemical nature of lead peroxide.

(*d*) **Lead Trioxide.** Place 5 c.c. sodium hypochlorite solution in a test tube and add 1 c.c. sodium hydroxide solution. To this mixture add lead nitrate solution until a permanent yellow precipitate is formed. What is this? Now add .5 c.c. concentrated nitric acid. If the precipitate dissolves, add sodium hydroxide solution until it reappears. Warm the solution. What is the brown precipitate which results?

(*e*) **Red Lead or Minium.** 1. Describe a s ecimen of red lead. Heat a little in a glass tube. Notice the color change. Is oxygen given off?

2. To a little minium in a test tube, add dilute nitric acid. Filter off the precipitate. What is it? Test the filtrate for lead. What is the chemical character of minium?

Experiment 155. Halogen Compounds of Lead. ¶ **205.** (*a*) **Lead Chloride.** Add dilute hydrochloric acid to a solution of lead nitrate. Heat the mixture to boiling, filter, and set it aside to crystallize.

(*b*) Perform a similar experiment with potassium bromide and lead nitrate solutions.

(*c*) Make lead iodide from potassium iodide and lead nitrate. Recrystallize the lead iodide from dilute acetic acid.

Experiment 156. Other Lead Salts. ¶ 205. (*a*) **Lead Nitrate.** Dissolve about 2 g. of granular lead in dilute nitric acid. Filter the solution, evaporate it, and set it aside to crystallize.

(*b*) **Lead Sulphate.** Add dilute sulphuric acid to a solution of lead nitrate. Test the solubility of the precipitate in alkalis and in ammonium acetate solution.

(*c*) **Lead Carbonate.** Add ammonium carbonate to a dilute solution of lead nitrate.

(*d*) **Lead Sulphide.** Pass hydrogen sulphide into dilute lead nitrate solution. Test the solubility of lead sulphide in acids and in alkalis.

ATOMIC WEIGHTS

¶ 208

Experiment 157. Combining Weight of Zinc. ¶ 208. Fill a liter flask with water that has attained the temperature of the room and invert it in a trough as in Fig. 42. Fit a wide-mouthed 100-c.c. flask with a two-holed rubber stopper carrying a dropping funnel, and with a long exit tube bent so as to open into the mouth of the liter flask in the trough. Weigh accurately 2 g. of chemically pure zinc, and place it in the 100-c.c. flask. Fill the flask, exit tube, and the stem of the funnel tube completely with water. Place the apparatus so that the mouth of the exit tube is under the mouth of the liter flask. Fill the funnel tube with concentrated hydrochloric acid. Allow the acid to enter the flask in small portions so that hydrogen is generated in a steady stream. Allow the apparatus to stand until the zinc is completely consumed. Then drive all the gas into the liter flask by means of water from the dropping funnel.

Allow the gas to attain the temperature of the room. Take the thermometer and barometer readings. Lower the flask into the water so that the level is the same outside and inside. Keeping the level the same, close the flask with the palm of the hand and remove it from the trough. After wiping the flask dry, weigh it to 1 g. Fill the flask completely with water and weigh it again.

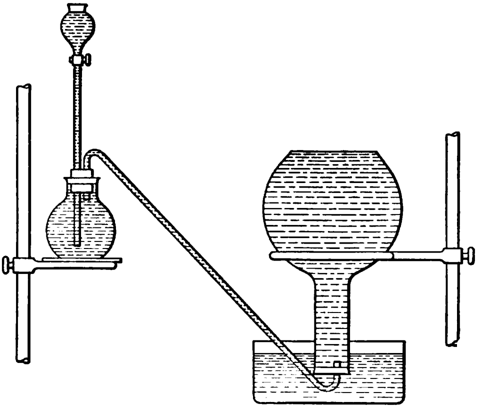

FIG. 42.

Make your calculations as follows:

Weight of the zinc.................. = ...2 g.
Weight of the flask partly full of water..= a = ...g.
Weight of the flask full of water.......= b = ...g.
Volume of the hydrogen..............= $b-a$ = ...c.c.
Barometer reading...................= P = ...mm.
Temperature.........................= t = ...°
Pressure of water vapor at $t°$..........= h = ...mm.

Weight of Hydrogen =

$$\frac{273\times.0000899(b-a)(P-h)}{760(273+t)}=m=\ldots\ldots\text{g.}$$

You have found by experiment that m g. of hydrogen are replaced by 2 g. of zinc. Calculate how many grams of zinc would replace 1 g. of hydrogen.

SPECIFIC HEAT OF ZINC

¶ 208

Experiment 158. Weigh out 100 g. zinc pellets or zinc cut up into small pieces. Place the zinc in a tin cylinder about 3 cm. in diameter and 10 cm. high, fitted with a wire handle.

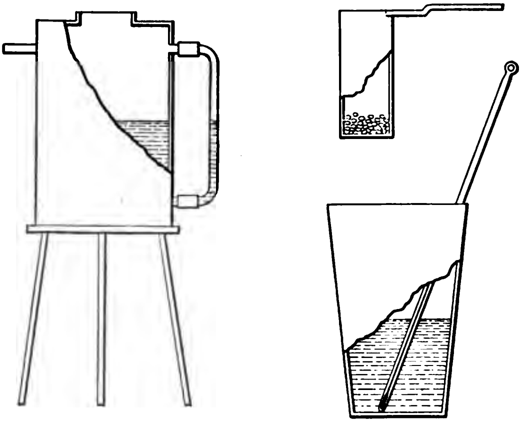

FIG. 43.

Heat the cylinder in a beaker of boiling water (or in a special water boiler made to fit the cylinder) until the zinc attains a constant temperature as shown by a thermometer placed in the zinc and with which the zinc is stirred. Weigh out 200 g. water into a weighed nickel-plated lemonade shaker or similar vessel and cool the water to about 10° below the temperature of the room. Transfer the zinc quickly to the water in the calorimeter (lemonade shaker), stir the mixture, and note the temperature as soon as it becomes constant.

Make your calculations as follows:

Weight of the calorimeter........................ = a = ...g.
Weight of the water......................... =200 g.
Temperature of water........................ = t° = ...°
Weight of zinc............................ =100 g.
Temperature of zinc........................ = t'° = ...°
Temperature of the mixture.................. = t''° = ...°
Specific heat of brass........................ =....094
Specific heat of zinc......................... =.... x.

The heat lost by the zinc equals the heat gained by the water and by the calorimeter (neglecting the thermometer), i. e.,

$$100x(t'-t'')=200(t''-t)+.094a(t''-t).$$

Calculate the value of x.

Calculation of the Atomic Weight of Zinc. From the Law of Dulong and Petit we know that

$$\text{At. Wt.}=\frac{6.4}{\text{Sp. Ht.}}.$$

Therefore

$$\text{At. Wt. of Zinc}=\frac{6.4}{\text{Sp. Ht. Zinc}}=\ldots\ldots$$

In Exp. 157, the combining weight of zinc was found. What, according to Exp. 157 and Exp. 158, is the value found by you for the atomic weight of zinc?

Examples. 1. The combining number for antimony is 40.66, and the specific heat is .051 cal. Calculate the exact atomic weight of antimony.

2. The combining weight of iron is 27.95 and the specific heat is .114 cal. Calculate the atomic weight of iron.

3. The combining weight of bismuth is 69.5, and the specific heat is .03 cal. Calculate the atomic weight of bismuth.

LITHIUM

¶ 222

Experiment 159. (*a*) **Lithium Hydroxide.** ¶ **222.** Heat a few crystals of lithium nitrate to redness on a piece of platinum foil. When the mass is cool, test it with moist red litmus paper. Dissolve the oxide in water, and add the solution to a solution of ferric chloride in a test tube.

(*b*) **Other Salts of Lithium.** 1. **Lithium Chloride.** Examine lithium chloride.

2. **Lithium Phosphate.** To 1 c.c. of a concentrated solution of a lithium salt alkaline with sodium hydroxide, add a strong solution of sodium phosphate.

(*c*) **Lithium Flame.** Examine the lithium flame by means of a small spectroscope. Use lithium chloride. See ¶ **263.**

SODIUM

¶¶ 223–226

Experiment 160. (*a*) 1. **Properties.** Examine a specimen of sodium and record its properties.

2. Support a 5-mm. glass tube about 10 cm. long perpendicularly so that one end dips under water. Drop a 2-mm. piece of sodium into the tube and ignite the hydrogen which escapes. See Fig. 44.

(*b*) **Sodium Peroxide.** Allow water to drop from a dropping funnel upon sodium peroxide in a generating flask. Collect the gas. What is it?

(*c*) **Sodium Chloride.** Pass hydrogen chloride into a concentrated solution of sodium chloride. Explain the precipitation of the salt.

(*d*) **Sodium Hydroxide.** To a strong solution of sodium carbonate in a beaker, add an excess of slaked lime, and boil the mixture for a few minutes. Filter the liquid, and evaporate it to dryness. Explain the formation of sodium hydroxide.

(*e*) **Sodium Sulphate.** Calculate the amount of concentrated sulphuric acid necessary to transform 10 g. of sodium chloride into sodium sulphate, and heat the mixture under the hood. Dissolve the residue in as little hot water as possible, and set the solution aside to crystallize. Examine and describe the product.

(*f*) **Le Blanc Process for Sodium Carbonate.** Powder 2.5 g. anhydrous sodium sulphate, 1.8 g. calcium carbonate, .2 g. calcium oxide, and 2 g. charcoal. Mix the substances intimately, and heat the mixture in an iron crucible with the blast lamp flame for about ten minutes, keeping the crucible covered. When the black mass is cool, remove it from the crucible, pulverize and warm it with about 10 c.c. of water. Filter the solution and set it aside to crystallize. What are the crystals? Test a little of the solution with hydrochloric acid. Test the solution with red litmus paper. Leave a crystal in the air for a day.

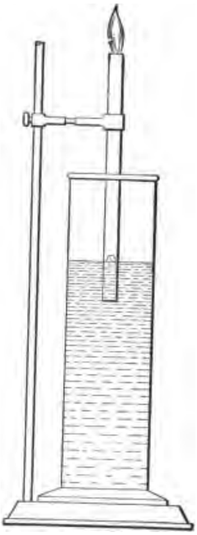

FIG. 44.

(*g*) **Solvay Process for Sodium Carbonate.** Make a saturated cold solution of sodium chloride (100 c.c.) Pass ammonia and carbon dioxide into the mixture. Cool the solution. Filter off the crystals. Are they sodium carbonate?

(*h*) **Sodium Silicate.** Fuse in an iron crucible a few grams of powdered quartz with the calculated amount of anhydrous sodium carbonate. When the melt is cool, dissolve it in water, and filter. What does the solution contain? To a little of the solution add concentrated hydrochloric acid. What is precipitated?

POTASSIUM

¶¶ 227–231

Experiment 161. (*a*) **Properties of Potassium.** Examine a specimen of the element potassium, and state its properties.

(*b*) **Potassium Hydroxide.** How could you make potassium hydroxide from potassium carbonate?

(*c*) **Potassium Bromide.** Calculate the amount of bromine necessary to neutralize 1 g. of potassium hydroxide. Dissolve the hydroxide in water and add a little more than the required amount of bromine. Evaporate the solution to dryness under the hood, add an excess of powdered charcoal to the powdered salt, and mix the mass thoroughly. Heat the mixture in a porcelain crucible, dissolve the cold product in hot water, filter the solution, and allow the filtrate to crystallize.

(*d*) **Potassium Iodide.** Calculate the amount of iodine required to form Fe_3I_8 with .5 g. of iron filings. Mix the iron and the required amount of iodine under 6 c.c. water until all is dissolved. Then add a strong solution of potassium carbonate so long as carbon dioxide escapes. Filter off the recipitate, evaporate the filtrate on the water-bath, and set the concentrated solution aside to crystallize. Describe the product.

(*e*) **Potassium Cyanide.** Fuse 3.5 g. of potassium carbonate with 10.5 g. of potassium ferrocyanide in an iron crucible. Dissolve the cooled mass in a small amount of hot water, filter and evaporate the solution to dryness under the hood.

(*f*) **Potassium Chlorate.** Pass chlorine into a boiling solution of potassium hydroxide under the hood until the alkaline reaction disappears. Evaporate the solution and allow it to crystallize.

(*g*) **Potassium Perchlorate.** Heat a few grams of potassium chlorate in a hard glass tube until the molten liquid which first forms ceases to give off oxygen and begins to grow semi-solid.

Allow the mass to cool, dissolve it in hot water, and allow it to crystallize.

(*h*) **Potassium Bisulphate.** Heat a little solid potassium bisulphate in a test tube, and describe the changes which take place.

(*i*) **Potassium Nitrate.** Make boiling saturated solutions of 10 g. of potassium chloride and 11.5 g. of sodium nitrate. Mix the solutions. What are the crystals which form when the solution cools?

(*k*) **Flame Reaction.** Test for the color of the potassium flame, using potassium chloride.

AMMONIUM SALTS

¶ 234

Review Experiment 87.

Experiment 162. (*a*) **Ammonium Phosphates.** 1. **Tertiary Salt.** Neutralize a concentrated solution of phosphoric acid with ammonia, and allow the mixture to crystallize.

2. **Secondary Salt.** Dry the crystals obtained in 1. Does ammonia escape?

3. **Primary Salt.** Dissolve the dried crystals from 2, in a small amount of hot water, boil the solution, and set it aside to crystallize.

4. **Microcosmic Salt.** Make a loop at the end of a platinum wire, and by means of this, heat a crystal of microcosmic salt in the Bunsen flame. What is given off? Heat the salt until a clear bead is obtained. Of what is the bead composed? What colors do various oxides such as cobalt, copper and manganese give when dissolved in the microcosmic bead and heated in the outer flame of the Bunsen burner?

(*b*) **Ammonium Carbonate.** In a test tube, heat a mixture of calcium carbonate and solid ammonium chloride. What distils off?

(*c*) **Ammonium Sulphide.** Saturate 10 c.c. of concentrated ammonia with hydrogen sulphide. Add an equal volume of ammonia. Dilute the solution and try its action upon solutions of salts of lead, copper, antimony, iron, nickel, etc. Dissolve a little sulphur in an ammonium sulphide solution. What is the product?

SALT SOLUTIONS

¶¶ 235–239

Experiment 163. Solubility Curves. From the following data, using co-ordinate paper, plot the solubility curve. Plot temperatures as abscissæ, and grams dissolved as ordinates.

Temperatures.	No. Grams in 100 g. Water.
0	5
10	10
20	20.1
30	40
32.5	47.5
33	51.5
35	50.25
40	49.5
50	47.5
60	45
70	44.75
80	44.65
90	44.65
100	44.65

Explain the signification of the curve obtained.

Experiment 164. Melting Point Curves. Two elements, *A* and *B*, are melted together in varying atomic proportions and the freezing points of the mixtures determined. From the following data, plot the melting point curve, plotting atomic percentages of *B* from left to right as abscissæ, and temperatures as ordinates.

Per cent of B.	Freezing Point. Degrees.
0	38
5	35.5
10	32.25
20	24.75
25	20
30	14.5
31	13
32	17
35	25.25
40	35.5
45	44
50	50
53	52.5
55	54
57	54.75
58	55
59	55.25
60	55.5
61	55.25
64	55
67	53
70	50.1
75	43.9
80	34.5
82	30
84	25
86	34
90	47.9
95	59.5
100	68.5

State, in detail, the signification of the points at which the curve changes direction, and what crystals separate out along each curve going downward and what the composition of the remaining liquid is.

ACIDIMETRY AND ALKALIMETRY

¶¶ 240, 241

Examples. 1. The standard of a certain hydrochloric acid solution is desired. One g. of pure dry sodium carbonate required 42.5 c.c. of the acid solution to neutralize it. Calculate the value of 1 c.c. of the solution in grams of hydrochloric acid. Express the strength of the solution in terms normal.

2. 15.4 c.c. of the hydrochloric acid solution in Ex. 1, were required to neutralize 25 c.c. of a solution of sodium hydroxide. Calculate the strength of the sodium hydroxide solution.

COPPER

¶¶ 242–244

Experiment 165. (*a*) **Occurrence.** Examine and describe specimens of malachite, azurite, chalcopyrite, and any other copper minerals that may be at hand.

(*b*) **Chemical Properties of Copper.** Review Exp. 76 (*a*) and Exp. 92 (*a*). Try the action of hydrochloric acid on copper. Place a nail in a solution of a copper salt.

(*c*) **Cuprous Compounds.** 1. **Cuprous Oxide.** To a Fehling's solution (a mixture of an alkaline solution of Rochelle salts and a solution of copper sulphate), add a solution of grape sugar, and boil the mixture until cuprous oxide is precipitated.

2. **Cuprous Chloride.** Try the solubility of cuprous chloride in concentrated hydrochloric acid and in strong ammonia.

Use small amounts in test tubes. For what purpose are similar solutions used in analysis?

3. **Cuprous Iodide.** To a solution of copper sulphate, add a solution of potassium iodide. Explain the formation of free iodine. What is the precipitate?

4. **Cuprous Cyanide.** To a copper sulphate solution in a test tube, add a solution of potassium cyanide. What is the precipitate? Now add an excess of the potassium cyanide. Explain the solubility of cuprous cyanide in potassium cyanide. Try to precipitate the copper from the solution by means of hydrogen sulphide. Explain.

(*d*) **Cupric Compounds.** 1. **Cupric Oxide.** In a hard glass tube, place a mixture of copper oxide and a small amount of sugar and heat the tube to redness. Explain the reduction.

2. **Cupric Chloride.** Dissolve a little cupric oxide powder in concentrated hydrochloric acid, and evaporate the solution to dryness. Heat the cupric chloride in a test tube so that the water of crystallization is driven off. What is the color of the anhydrous salt? Make a very concentrated solution of cupric chloride in a test tube. Then dilute the solution. Explain the color changes.

3. **Cupric Bromide.** Review Exp. 65 (*c*).

4. **Copper Sulphate.** Dehydrate powdered copper sulphate by heating it in a test tube. Using the anhydrous salt thus obtained, test ordinary alcohol for the presence of water.

5. **Basic Copper Carbonate.** Add a little sodium carbonate solution to a copper sulphate solution.

6. **Copper Sulphide.** Review Exp. 74 (*a*) and (*b*).

7. **Copper Salts and Ammonia.** To a solution of copper sulphate add an excess of ammonia. Compare the blue color thus obtained with the blue of a copper sulphate solution. Test the ammonia solution with dilute sodium hydroxide. Test similarly a copper sulphate solution. Why do the solutions act differently?

SILVER

¶¶ 245–247

Experiment 166. (*a*) **Occurrence.** Examine and describe any silver minerals that may be at hand.

(*b*) **Physical and Chemical Properties.** 1. Melt a small piece of silver foil on charcoal.

2. Try the solubility of silver in the mineral acids.

(*c*) **Compounds of Silver.** 1. **Silver Oxide.** To dilute silver nitrate solution, add a little dilute sodium hydroxide.*

2. **Silver Chloride.** Precipitate silver chloride from dilute silver nitrate by means of hydrochloric acid. Allow a little of the precipitate to stand in the light. Try the solubility of the precipitate in ammonia, in sodium thiosulphate, and in potassium cyanide solutions.

3. **Silver Bromide and Iodide.** Similarly precipitate silver bromide and iodide.

4. **Silver Cyanide.** To a dilute solution of silver nitrate in a 100-c.c. beaker, add potassium cyanide until the precipitate first formed dissolves. Cut two small strips, one of copper and the other of silver foil, and attach copper wires to them. Place the metal strips in the solution and connect them with an electric cell so that the copper is the cathode and the silver is the anode. Explain the formation of the silver deposit on the copper and the corrosion of the silver strip.

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* Put all silver precipitates and silver solutions in the bottle marked "Silver Residues."

GOLD

¶ 248

Experiment 167. (*a*) **Occurrence.** Examine and describe any gold minerals that may be at hand, such as free gold, calaverite, etc.

(*b*) **Physical and Chemical Properties.** State the physical properties of gold as far as you know them. Examine a piece of gold leaf by transmitted light. Try the effect of chlorine water, aqua regia, and potassium cyanide solution on gold leaf.

(*c*) **Purple of Cassius.** To 50 c.c. of water, add 1 drop of stannous chloride solution, and 2 drops of sodium hydroxide solution. Add this solution slowly, drop by drop, to 5 c.c. of a .01 per cent auric chloride solution.

(*d*) **Precipitation of Gold.** To 5 c.c. of a .01 per cent auric chloride solution, add a few drops of ferrous sulphate, and note the color of the solution by reflected and by transmitted light.

MAGNESIUM

¶¶ 254, 255

Experiment 168. (*a*) **Occurrence.** Examine and describe specimens of magnesite, talc, asbestos, meerschaum, and any other magnesium minerals that may be at hand.

(*b*) **Physical and Chemical Properties.** Clean a piece of magnesium wire and examine the metal as to luster, hardness, etc. Burn a piece in the Bunsen flame. Try the action of very dilute acids upon magnesium.

(*c*) **Compounds of Magnesium.** 1. **Magnesium Oxide and Hydroxide.** Test with moist red litmus paper the white oxide obtained in (*b*). Test strong magnesium sulphate solution with a little dilute ammonia. What is the precipitate? Similarly test a solution of magnesium sulphate to which an excess of ammonium chloride has been added. Explain why a precipitate is not formed.

2. **Magnesium Chloride.** Boil solid magnesium chloride with water in a test tube, testing the boiling solution with blue litmus paper. Explain the acid reaction.

3. **Magnesium Ammonium Phosphate.** Add a solution of acid sodium phosphate to an ammoniacal solution of a magnesium salt to which ammonium chloride has been added. Examine the precipitate with a magnifying glass. See Exp. 109 (*b*) 5.

4. **Basic Magnesium Carbonate.** To a dilute solution of a magnesium salt, add dilute sodium carbonate.

CALCIUM

¶¶ 256–260

Experiment 169. (*a*) **Occurrence.** Examine and describe specimens of limestone, calcite, aragonite, marble, phosphorite, gypsum, etc.

(*b*) **Properties.** Examine a small piece of electrolytic calcium, as to luster, hardness, etc. Try the effect of water upon a small piece. After the action has been noted, add dilute hydrochloric acid.

Experiment 170. Compounds of Calcium. (*a*) **Calcium Oxide. Calcium Hydroxide.** Heat a small piece of marble in a hard glass tube to a red heat in the blast lamp flame. Remove the cooled substance from the tube and treat it with a few drops of water. Does it affect moist red litmus paper?

(*b*) **Calcium Chloride.** 1. **Dehydrating Power.** Shake about 5 c.c. benzene with a drop of water. Now add a few pieces of anhydrous calcium chloride, and allow the mixture to stand.

2. **Freezing Mixture.** To about 20 g. of crushed ice add as much crystallized calcium chloride and mix the two intimately. Take the temperature of the mixture. Explain why a temperature no lower than −48.5° may be obtained by this method.

(*c*) **"Chloride of Lime."** 1. **Action with Acids.** To "chloride of lime," add a little hydrochloric acid. What is evolved?

2. **Bleaching Action.** Place a piece of colored paper in water containing "chloride of lime," and add dilute acetic acid.

(*d*) **Calcium Sulphate.** Make a paste of some "Plaster of Paris," and take an impression of some object. Explain the "setting" of "Plaster of Paris."

(*e*) **Calcium Phosphate.** To a solution of a calcium salt, add a solution of acid sodium phosphate. What is "superphosphate"?

(*f*) **Calcium Carbonate and Acid Calcium Carbonate.** Pass carbon dioxide through a solution of lime water until the precipitate first formed (what is it?) is dissolved (what soluble compound is formed?). Now boil part of the solution until a white precipitate appears. Explain. To the remainder of the solution, add a drop of a soap solution, and shake the mixture. Does a lather form? Add soap solution drop by drop until a lather forms. Explain. What is temporarily hard water? How can it be made soft?

(*g*) **Calcium Oxalate.** To an ammoniacal solution of calcium chloride, add a solution of ammonium oxalate. Try the solubility of the precipitate in acetic acid and in hydrochloric acid. Explain why it does not dissolve in acetic, but is soluble in hydrochloric acid.

(*h*) **Glass.** 1. Fuse by means of a blowpipe flame upon charcoal a small amount of a mixture of 2 parts of potassium carbonate, 1 part of silica, and 1 part lead oxide.

2. **Action of Water upon Glass.** Shake a little powdered glass with water. Then add a drop of phenolphthalein solution. Explain.

STRONTIUM

¶ 261

Experiment 171. (*a*) **Occurrence.** Examine and describe specimens of strontianite and celestite.

(*b*) **Strontium Compounds.** 1. **Strontium Hydroxide.** Add dilute sodium hydroxide to a strontium chloride solution.

2. **Strontium Sulphate.** Add dilute sulphuric acid to a strontium chloride solution.

3. **Strontium Flame.** Observe the strontium flame by holding a platinum wire moistened with strontium chloride in the Bunsen flame.

BARIUM

¶ 262

Experiment 172. (*a*) **Occurrence.** Examine and describe specimens of barite and witherite.

b. **Barium Compounds.** 1. **Barium Hydroxide.** Test a solution of barium hydroxide with red litmus paper. Pass carbon dioxide into a solution of barium hydroxide.

2. **Barium Peroxide. See Exp. 43** (*a*).

3. **Barium Sulphate.** Add dilute sulphuric acid to a barium chloride solution. Test the solubility of the precipitate in the mineral acids.

4. **Barium Flame.** Using barium chloride, observe the barium flame.

ZINC

¶¶ 268, 269

Experiment 173. (*a*) **Occurrence.** Examine and describe specimens of calamine, smithsonite, and sphalerite.

(*b*) **Properties of Zinc.** 1. Heat a piece of zinc in a porcelain crucible by means of the blast lamp flame, so that the zinc burns to the oxide. Describe the appearance of the oxide. Note the change of color on heating.

2. Try the action of the mineral acids upon zinc.

3. Try the effect of a strip of zinc upon a copper solution. Explain.

(*c*) **Compounds of Zinc.** 1. **Zinc Oxide.** See (*b*) 1.

2. **Zinc Hydroxide.** Add dilute sodium hydroxide to a zinc sulphate solution. Now add more sodium hydroxide until the precipitate dissolves. Repeat the experiment, using ammonia instead of sodium hydroxide. Explain.

3. **Zinc Sulphide.** Pass hydrogen sulphide through a solution of zinc chloride, slightly acid with hydrochloric acid. Does a precipitate form? Now add some crystals of sodium acetate, and pass hydrogen sulphide through the solution again. Explain the formation of the white precipitate. To another solution of zinc chloride, add ammonium sulphide solution. Explain the formation of the white precipitate.

CADMIUM

¶ 270

Experiment 174. (*a*) **Properties.** 1. Examine a piece of cadmium, and compare it with zinc as to its physical properties.

2. Heat a piece of cadmium on charcoal by means of the blowpipe flame.

3. Try the solubility of cadmium in the mineral acids.

(*b*) **Cadmium Compounds.** 1. **Cadmium Oxide.** Note the incrustation formed on the charcoal in (*a*) 2.

2. **Cadmium Hydroxide.** To a dilute solution of cadmium chloride, add dilute sodium hydroxide. Is cadmium hydroxide acid or basic? How do you know?

3. **Cadmium Sulphide.** Pass a stream of hydrogen sulphide through a dilute, slightly acid solution of cadmium chloride. Does cadmium sulphide act as an acid toward an alkali sulphide? How do you know?

MERCURY

¶¶ 271–274

Experiment 175. (*a*) **Occurrence.** Examine and describe the mineral cinnabar.

(*b*) **Properties.** Bring a little gold leaf in contact with a drop of mercury. Clean a piece of copper, and dip it first into nitric acid and then into a solution of a mercury salt.

(*c*) **Mercurous Compounds.** 1. **Mercurous Oxide.** Add dilute sodium hydroxide to a solution of mercurous nitrate. Boil the mixture.

2. **Mercurous Chloride.** Add dilute hydrochloric acid to dilute mercurous nitrate in a test tube. Then add a little ammonia.

3. **Mercurous Bromide and Iodide.** Make mercurous bromide and mercurous iodide in a test tube.

4. **Basic Mercurous Nitrate.** Add 3 c.c. of water to a few crystals of mercurous nitrate in a test tube. Boil the mixture. To what is the yellow color due?

(*d*) **Mercuric Compounds.** 1. **Mercuric Oxide. See Exp. 12** (*a*). Add dilute sodium hydroxide to a solution of mercuric nitrate or chloride.

2. **Mercuric Chloride.** Compare mercurous and mercuric chloride as to appearance and solubility.

Shake a solution of common salt with mercuric oxide. Explain the alkaline reaction.

3. **Mercuric Iodide.** To a solution of mercuric chloride, add a solution of potassium iodide.

4. **Nessler's Solution.** Continue adding potassium iodide to the solution obtained in (*d*) 3, until the red precipitate dissolves. Then make the solution strongly alkaline with potassium hydroxide. Add 2 c.c. of the solution to a dilute solution of ammonium chloride. What does the yellow coloration indicate?

ELECTROCHEMISTRY

¶ 276

Experiment 176. Faraday's Law. Cut two pieces of copper foil 5 cm. by 8 cm. Solder a copper wire to each. Clean the plates with sandpaper, dip them in concentrated nitric acid, rinse them with distilled water, dry one of them and weigh it to .01 g. Place the copper plates in an approximately normal solution of copper sulphate acid with sulphuric acid. Connect one of the copper plates with one electrode of a Hofmann's apparatus for the electrolysis of water containing dilute sulphuric acid (1 : 12). Pass a current from 5 to 10 accumulators through the apparatus so that the weighed copper plate is the cathode. Continue the electrolysis until about 50 c.c. hydrogen have been obtained. Remove the copper electrode while the current is still passing, wash it with distilled water, then with alcohol, dry and weigh it to .01 g. When the hydrogen has cooled to the temperature of the room, read the volume. Take the temperature and the barometer reading. Measure the height of the water column supported by the hydrogen in the apparatus. Calculate the weight of the hydrogen, correcting the barometer reading for the tension of water vapor and for the height of the water column. Compare the weight of hydrogen obtained with the weight of copper deposited. Calculate how many grams of copper were deposited for one gram of hydrogen.

State Faraday's Law.

Experiment 177. The Daniell Cell. Set up a Cu-Zn Daniell cell as follows: Half fill a 100-c.c. beaker with about normal copper sulphate solution. Fill a porous porcelain cylinder with about normal zinc sulphate solution. Make electrodes of copper and zinc, place them in their solutions, and put the whole in circuit with a galvanoscope. Note the movement of the needle. Under a hood add strong potassium cyanide solution to the copper sulphate beaker until the solution is decolorized. Note the movement of the needle. Explain the phenomena in the light of Nernst's theory.

Experiment 178. The Lead Accumulator. Cut two strips of lead sheet about 2 by 4 cm. and scrape them clean. Place them in a beaker containing sulphuric acid (1 : 1). Pass a current from two accumulators or bichromate cells through the acid, noting the direction of the current through the cell. After a few minutes disconnect the cell and place it in circuit with a galvanoscope. Do you get a current? In what direction? Explain the source of the current.

Experiment 179. ¶ 277. Transformation of Chemical Energy into Electrical Energy. Apply to your instructor for a Lüpke cell. In one vessel place an acidulated normal solution of sodium chloride, in the second an acidulated solution of stannous chloride, and connect the two by means of a siphon filled with the sodium chloride solution. Place the cell in circuit with a galvanoscope. Is a current indicated by the instrument? Now allow a few drops of chlorine water to flow from a pipette upon the electrode in the sodium chloride solution. Explain the source of the current which is indicated by the instrument.

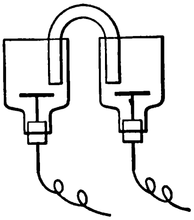

FIG. 45.

Experiment 180. The Oxygen-Hydrogen Gas Cell. Obtain from your instructor a gas cell. Fill the cell with dilute sulphuric acid, and the tubes, one with hydrogen and the other with oxygen. Place the cell in circuit with a galvanoscope. Explain the source of the current which is indicated by the instrument.

Experiment 181. Electrolytic Corrosion of a Metal. Place clean rods of iron and tin in a very dilute solution of sodium chloride, through which air and carbon dioxide are passing, and connect the rods with a galvanoscope. Is a current produced? After a few minutes, note the coating of rust on the iron rod. Which has the greater electrolytic solution pressure, iron or tin? Why does tin plate rust very fast on a spot where the tin covering has been removed?

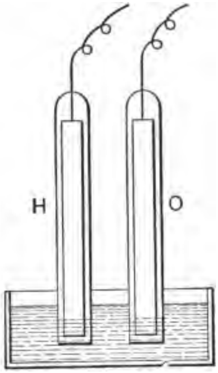

Fig. 46.

BORON

¶¶ 282, 283

Experiment 182. (*a*) **Occurrence.** Examine and describe any boron minerals that may be at hand.

(*b*) **Properties.** 1. Examine and describe a specimen of amorphous boron.

(*b*) **Oxygen Compounds of Boron.** 1. **Boric Anhydride.** Heat a little boric acid on a platinum wire loop. What is the glassy mass?

2. **Boric Acid.** Dissolve 1 g. of boric acid in sodium hydroxide solution and evaporate the salt solution on the water-bath. What is the product?

(*c*) **Borax.** 1. **Solubility of Metallic Oxides in Borax Glass.** Perform an experiment similar to Exp. 162 (*a*), 4, using borax instead of microcosmic salt.

2. **Hydrolysis of Borax.** To a concentrated solution of borax, add litmus solution and then acetic acid until the red color just appears. Dilute the solution. Explain the appearance of the blue color.

ALUMINUM

¶¶ 284–286

Experiment 183. (*a*) **Occurrence.** Examine and describe specimens of corundum, bauxite, feldspar, mica, cryolite, kaolin, etc.

(*b*) **Properties.** 1. Examine a specimen of aluminum metal, and state its physical properties.

2. Try the effect of the various acids upon aluminum. Try the effect of alkalis.

5. **Thermite.** Place about 10 c.c. commercial "thermite" in a clay crucible, and cover it with a thin layer of "ignition mixture" (a mixture of barium peroxide and magnesium powder). Place an "igniter" upon the mixture. By means of a match held in a pair of tongs, ignite the magnesium ribbon. Explain the vigorous reaction which takes place.

4. **Aluminum Amalgam.** Add aluminum filings to a ½ per cent solution of mercuric chloride. What is formed? Try its effect upon a dilute neutral solution of potassium permanganate.

(*b*) **Compounds of Aluminum.** ¶ **285.** 1. **Aluminum Hydroxide.** Add dilute sodium hydroxide to a dilute solution of an aluminum salt. Divide the precipitate into two portions. To one add an excess of the alkali, and to the other add hydrochloric acid. Is aluminum hydroxide strongly basic? Strongly acid?

2. **Aluminum Chloride.** Examine a specimen of anhydrous aluminum chloride. How was the substance made? Examine a specimen of the hydrated salt.

3. **Aluminum Sulphate.** Heat about 2 g. of powdered white clay with concentrated sulphuric acid in a 150-c.c. flask. After five minutes, cool the liquid, dilute, filter and evaporate it to crystallization.

4. **Alums.** Dissolve molecular amounts of aluminum sulphate and potassium sulphate in the least possible amount of hot water, and set the solution aside to crystallize. When crystals have separated out, note their form. Remove a crystal from the solution and heat it in the flame. Taste the substance. Define an " alum."

Write on a glass plate with an alum crystal, and pour a concentrated alum solution upon it.

CHROMIUM

¶¶ 293–295

Experiment 184. (*a*) **Occurrence.** Examine and describe specimens of chromite and crocoite.

(*b*) **Chromous Compounds.** 1. **Chromous Chloride.** Reduce a solution of chromic chloride with zinc and sulphuric acid, keeping the mouth of the test tube loosely stoppered to exclude air.

2. **Chromous Acetate.** Pour a concentrated solution of sodium acetate into the solution obtained in 1.

(*c*) **Chromic Compounds.** **¶ 294.** 1. **Hydrogel of Chromic Oxide.** Add ammonium hydroxide solution to a dilute solution of chromic sulphate. Test the solubility in excess of the alkali.

2. **Chromic Chloride.** Compare the anhydrous variety of chromic chloride with that which contains water of crystallization.

3. **Chromic Sulphate.** Dissolve a few crystals of potassium chromium sulphate in cold water. Observe the color of the solution by reflected and by transmitted light. Now heat the solution to boiling, and observe the color again. Explain the cause of the difference in color.

(*c*) **Chromates.** **¶ 299.** 1. **Chromic Anhydride.** Examine some crystals of chromic anhydride. Heat some crystals in a glass tube and test for oxygen. Let a drop of alcohol fall on a few crystals of chromic anhydride. Add a small amount of hydrochloric acid to chromic anhydride in a test tube. Is chlorine given off?

2. **Potassium Chromate.** Mix a little powdered chromite with solid sodium carbonate and potassium nitrate on a piece of platinum foil, and melt the mixture by the heat of the blowpipe flame. When the melt is cool, observe the color. Dissolve it in water, make acid with acetic acid, and add a lead acetate solution. What is the yellow precipitate?

3. **Potassium Bichromate.** Dissolve a few crystals of potassium chromate in a small amount of water, add dilute sulphuric acid, and evaporate to crystallization.

MOLYBDENUM

¶ 296

Experiment 185. (*a*) **Occurrence.** Examine and describe specimens of molybdenite and wulfenite.

(*b*) **Ammonium Molybdate.** This is the most common compound of molybdenum. See Exp. 109, (*b*), 6.

(*c*) **Characteristic Test for Molybdenum.** Treat the solution of the substance with zinc and dilute sulphuric acid. If molybdenum is present a blue coloration will appear, which soon turns green, and then brown.

TUNGSTEN

¶ 297

Experiment 186. (*a*) **Occurrence.** Examine specimens of scheelite, wolframite, and ferberite.

(*b*) **Tungstic Oxide.** Powder about 1 g. sodium tungstate and sprinkle it into boiling hydrochloric acid in a 100-c.c. beaker. The yellow substance is tungstic acid, H_2WO_4, which on being heated yields tungstic oxide, WO_3.

(*c*) **Characteristic Test for Tungsten.** Stannous chloride gives a yellow precipitate with a tungstate solution, which with hydrochloric acid yields a blue solution. A soluble tungstate yields a blue coloration with zinc and hydrochloric acid, the color changing gradually to brown.

URANIUM

¶ 298

Experiment 187. (*a*) **Occurrence.** Examine and describe a specimen of pitchblend or of uraninite.

(*b*) **Test for Uranyl Salts.** Treat a solution of a uranyl salt (1 c.c.) with potassium ferrocyanide. The brown precipitate is characteristic.

MANGANESE

¶¶ 300, 301

Experiment 188. (*a*) **Occurrence.** Examine and describe specimens of pyrolusite, hausmannite, and rhodochrosite.

(*b*) **Manganous Compounds.** 1. **Manganous Hydroxide.** Add ammonia to a solution of manganous chloride in a test tube. Note the color of the precipitate. Shake the precipitate. What causes the change in color? To a second solution of manganous chloride, first add ammonium chloride, and then ammonia. Why do you fail to get a precipitate of manganous hydroxide.

2. **Manganous Chloride and Sulphate.** Examine and describe specimens of these salts. Dissolve molecular proportions of manganous chloride and ammonium chloride in a small amount of hot water, filter the solution if necessary, and allow the double salt to crystallize out. Similarly prepare potassium manganous sulphate.

3. **Manganous Sulphide.** Precipitate manganous sulphide from a manganous solution by means of ammonium sulphide. Test its solubility in the ordinary acids.

(*c*) **Manganic Compounds. Manganese Dioxide.** Powder a small quantity of manganese dioxide and add cold concentrated hydrochloric acid to it in a test tube. Note the color of the solution. What is the solution supposed to contain? Heat the solution and note the evolution of chlorine. See Exp. 30 (*a*).

(*d*) **Manganates. ¶ 301.** Mix a small quantity of manganese dioxide powder with about five times as much of a mixture of dry sodium carbonate and potassium nitrate. Heat the mixture to complete fusion on a piece of platinum foil. When the mass is cold, note its color, and dissolve the mass in cold water. Warm the solution and note the color change. Explain the reactions.

(*e*) **Permanganates. ¶ 301. 1. Oxidizing Power of Potassium Permanganate in Acid Solution.** Add dilute sulphuric acid to a hot solution of oxalic acid in a beaker. Then add a small quantity of potassium permanganate solution. Explain.

2. **Oxidizing Action of Potassium Permanganate in Neutral or Alkaline Solution.** Add hot alcohol from a test tube to a beaker containing hot dilute potassium permanganate solution. What is the precipitate?

3. **Permanganic Anhydride. See Exp. 42** (*b*). What was the origin of the brown fumes, which were given off in Exp.

42 (*b*), when sulphuric acid was added to the potassium permanganate in the beakers?

IRON

¶¶ 302–308

Experiment 189. (*a*) **Occurrence.** Examine and describe specimens of hematite, limonite, siderite, and pyrite.

(*b*) **Extraction.** Draw a diagram of a modern blast furnace.

(*c*) **Properties of Iron.** 1. **Effect of Acids.** Try the effect of the mineral acids upon iron.

2. **Rusting.** Place a clean iron nail in a beaker with a little water, and examine the nail the next day. Similarly cover a clean nail with sodium hydroxide solution and examine it the next day.

3. **Passive Iron.** Cover a clean nail with concentrated nitric acid. Remove the nail, rinse off the acid, and again try the effect of nitric acid upon it.

4. **Modification of the Physical Properties of Iron by the Presence of Other Elements.** Make a table showing the effect upon iron of the presence of sulphur, silicon, carbon, phosphorus, nickel, chromium, tungsten, and vanadium.

5. **Steel.** Draw a diagram of a Bessemer converter.

(*d*) **Ferrous Compounds. ¶ 306.** 1. **Ferrous Hydroxide.** Add sodium hydroxide solution to a ferrous chloride solution in a test tube. Note how the precipitated ferrous hydroxide immediately begins to oxidize.

2. **Ferrous Chloride.** Dissolve a few iron nails in a 100-c.c. flask by means of hydrochloric acid (1 : 1). Filter the solution and evaporate it to crystallization. What are the crystals?

3. **Ferrous Sulphate.** Dissolve a few small nails in dilute sulphuric acid, evaporate the solution, and allow it to crystallize.

4. **Ferrous Carbonates. See Exp. 189** (*a*). Add sodium carbonate to a ferrous solution. Note how the precipitated basic carbonate oxidizes.

(*e*) **Ferric Compounds.** **¶ 307.** 1. **Ferric Oxide.** **See Exp. 189** (*a*).

2. **Ferric Hydroxide.** Add sodium hydroxide to a ferric solution in a test tube.

3. **Ferric Chloride.** Add chlorine water to a solution of ferrous chloride in a test tube. Shake the solution with a small quantity of ether. Is ferric chloride soluble in ether?

4. **Ferric Sulphate.** Dissolve a few crystals of ferrous ammonium sulphate in water. Pour a few drops of the solution into a test tube and add a small quantity of potassium sulphocyanate solution. Now add sulphuric acid and hydrogen peroxide to the original solution.

5. **Potassium Ferrocyanide.** Ignite under a good hood in an iron crucible 5 g. of blood albumen, adding the albumen a little at a time until all is charred. Mix about 10 g. of potassium carbonate and 10 g. of iron filings with the charred mass in the crucible, and ignite the mixture over the blast lamp flame. When the melt is cold, dissolve it in hot water, filter, and allow the solution to crystallize.

6. **Hydroferrocyanic Acid.** Add concentrated hydrochloric acid to a strong solution of potassium ferrocyanide in a test tube. Shake the tube. What causes the blue color?

7. **Sodium Nitroprusside.** Dilute one drop of sodium sulphide solution by adding 10 c.c. of water. Test the solution by adding a small amount of sodium nitroprusside solution.

8. **Potassium Ferricyanide.** Add an excess of bromine water to a solution of potassium ferrocyanide in a test tube. Evaporate the solution. What is formed?

Test a potassium ferricyanide solution with ferric chloride. Now add a solution of formaldehyde to an alkaline solution of potassium ferricyanide, boil it, and test it with ferric chloride Explain.

COBALT

¶¶ 309, 310

Experiment 190. (*a*) **Occurrence.** Examine and describe specimens of smaltite and cobaltite.

(*b*) **Cobaltous Compounds.** 1. **Cobaltous Chloride.** Examine some cobaltous chloride crystals. Dehydrate a crystal by heating it in a test tube.

2. **Cobalt Nitrate.** Heat a crystal of cobalt nitrate in a test tube until all brown fumes are given off. What is the black residue? Preserve for (*c*) 1.

3. **Thenard's Blue.** Moisten a lump of aluminum oxide with cobalt nitrate solution and ignite it strongly.

(*c*) **Cobaltic Compounds. ¶ 310.** 1. **Cobaltic Oxide.** Heat the cobaltic oxide obtained in (*b*) 2, with concentrated hydrochloric acid. Is chlorine given off?

2. **Potassium Cobalticyanide.** Add potassium cyanide to a cobaltous chloride solution until the brown precipitate first formed is dissolved. What does the solution contain? Now warm the solution until it becomes yellow. What does the solution now contain?

3. **Potassium Cobaltic Nitriet.** Make a cobalt nitrate solution acid with acetic acid, and add a small piece of solid potassium nitrite.

NICKEL

¶¶ 311, 312

Experiment 191. (*a*) **Occurrence.** Examine and describe specimens of niccolite, nickel glance, and garnierite.

(*b*) **Nickelous Compounds.** 1. **Nickel Chloride.** Dehydrate a crystal of nickel chloride in a test tube.

2. **Nickel Nitrate.** Heat a crystal of nickel nitrate in a test tube until no more brown fumes come off. What is the black residue?

3. **Nickel Sulphide. ¶ 312.** Precipitate nickel sulphide with ammonium sulphide, and test its solubility in acids.

4. **Potassium Nickel Cyanide.** Add potassium cyanide to a nickel solution until the green precipitate first formed is dissolved. What does the solution contain? Try the effect of hydrochloric acid upon the solution. Explain.

PLATINUM

¶ 316

Experiment 192. (*a*) **Properties of Platinum.** 1. Is platinum oxidized by heating in the air? Do the ordinary acids attack it? Does aqua regia attack it? Use short pieces of platinum wire in making these tests.

2. Heat the end of a platinum wire looped around a small piece of galena. Similarly test the effect of silicon on platinum.

(*b*) **Chlorplatinic Acid.** Test a solution of a potassium chloride with chlorplatinic acid. Similarly test a solution of ammonium chloride and one of sodium chloride.

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